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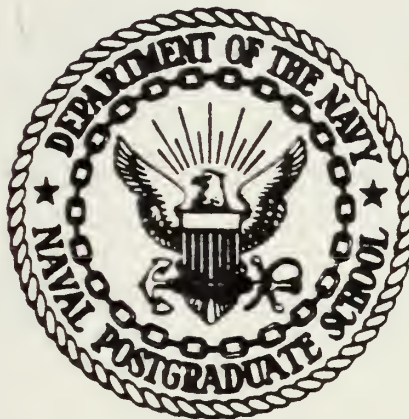
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THESIS

The Effect of Grain and Carbide Refinement
on the Isothermal Transformation
Characteristics of AISI 52100 Steel

by

Duane M. Tufte

September 1981

Thesis Advisor:

T. R. McNelley

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) The Effect of Grain and Carbine Refinement on the Isothermal Transformation Characteristics of AISI 52100		5. TYPE OF REPORT & PERIOD COVERED Master's Thesis September 1981
7. AUTHOR(s) Duane M. Tufte		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Postgraduate School Monterey, California 93940		8. CONTRACT OR GRANT NUMBER(s)
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Postgraduate School Monterey, California 93940		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
12. REPORT DATE September 1981		13. NUMBER OF PAGES 74
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report)
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release, distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) 52100 Steel; Thermomechanical Processing; Isothermal Transformation; Bainite; Microstructural Refinement; Grain Size; Residual Carbides; Retained Austenite; Mechanical Properties; Hardness.		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A study of the microstructure and mechanical properties of isothermally-transformed AISI 52100 bearing steel was conducted. Standard spheroidized annealed and thermomechanically grain-refined materials were compared. Heat treatment consisted of austenitization at 850°C, followed by isothermal transformation at 350°C, 275°C or 250°C. Hardness, retained austenite, and optical and transmission electron microscopy data were obtained		

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The Effect of Grain and Carbide
Refinement on the Isothermal Transformation
Characteristics of AISI 52100 Steel

by

Duane M. Tufte
Lieutenant, United States Navy
B.S., University of Kansas, 1975

Submitted in partial fulfillment of the
requirements for the degree of

MASTER OF SCIENCE IN MECHANICAL ENGINEERING

from the

NAVAL POSTGRADUATE SCHOOL
September 1981

ABSTRACT

A study of the microstructure and mechanical properties of isothermally-transformed AISI 52100 bearing steel was conducted. Standard spheroidized annealed and thermo-mechanically grain-refined materials were compared. Heat treatment consisted of austenitization at 850°C, followed by isothermal transformation at 350°C, 275°C or 250°C. Hardness, retained austenite, and optical and transmission electron microscopy data were obtained as a function of isothermal transformation time. Tensile test results also were obtained on selected conditions. It was found that isothermal holding above M_s resulted in comparable hardness values for both materials. Retained austenite remained low for all cases but did tend to increase as the M_s was approached. Microstructural analysis revealed that the fine-grain structure of the refined material was retained throughout the transformations while little or no difference in the carbide structure was noted. Tensile results show that the refined and transformed material exhibits greater strength and ductility.

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ACKNOWLEDGEMENT

I wish to express my deep gratitude to Professor Terry McNelley, Professor Michael Edwards, Professor Donald Boone and Professor K. D. Challenger for their guidance and assistance. I would also like to thank the Defense Systems Division of Honeywell Inc. for their cooperation and assistance in the differential thermal analysis performed. In addition, I wish to express my deepest gratitude to my wife, Phyllis and our sons, Tom and Brent, without whose continuous support and patience this work would not have been possible.

I. INTRODUCTION

A. BACKGROUND

This research examined the effect of grain and carbide refinement on the isothermal transformation characteristics of AISI 52100 steel. This work is part of an ongoing effort at the Naval Postgraduate School (NPS), evaluating thermomechanical processing to refine microstructure and the mechanical and microstructural characteristics of such materials after heat treatment. Research at NPS by Chung [Ref. 1] was directed toward the effect of thermomechanical processing and subsequent heat treatment on the microstructural and fatigue properties of this steel. It was concluded that an isothermal warm rolling prior to a conventional heat treatment resulted in a fine ferrite grain size with a dispersion of fine carbides. These microstructural features were further determined to be factors in the enhancement of strength, toughness and fatigue life. Additional work done by McCauley [Ref. 2] found that a fine grain and carbide distribution was retained throughout various conventional and isothermal heat treatments. He also reported that the fracture toughness of these refined materials was improved. Although these previous endeavors are not specifically directed toward the isothermal heat treatment of AISI 52100 steel, the conclusions are significant.

This work follows the more recent research of Schultz [Ref. 3] which involved the characterization of the thermo-mechanical refinement of grain and carbide structure of 52100 steel. His research compared and evaluated the response of the refined material to various hardening and tempering treatments to that of material processed using standard commercial methods. Schultz [Ref. 3] observed that the thermomechanical treatments refine the microstructure and increase the tensile strength of this steel. He also reported that the use of these treatments allows hardening of the steel to be accomplished with lower austenitizing temperatures and shorter austenitizing times.

Sherby and his co-workers [Refs. 4,5] at Stanford University have investigated warm rolling as a process whereby a fine spheroidized structure is developed in high-carbon and ultra-high carbon steels. In steels, martensitic or pearlitic microstructures will spheroidize when heated to just under the eutectoid temperature. This spheroidization softens the steel by breaking down the martensite or the lamellar pearlite and results in a more stable grain structure. Sherby, et. al., [Refs. 4,5] have established that concurrent plastic deformation during this spheroidizing anneal dramatically increases the rate of spheroidization and results in fine, spheroidal carbides in a refined ferrite matrix. Although their research has focused on ultra-high carbon steels containing 1.3 to 2.1% carbon, the process has been

found to be applicable as well to AISI 52100 steel, and the NPS work has been primarily concerned with application of this method to the 1.0% carbon 52100 steel.

AISI 52100 steel was developed in the 1920's for use in anti-friction bearing components [Ref. 6]. Much research has been directed toward increasing the reliability of ball and roller bearings by increasing their fatigue life. These efforts have led to increased reliability in engine and aerospace equipment. Anderson and Zaretsky [Ref. 7], in a review of the current state of development of rolling element bearings, note that in support of these engines, as well as other high performance equipment, a reliable bearing system composed of three key items is required. These are: a suitable lubricant; a reliable bearing structural material; and an optimized bearing design coupled with proper operating parameters. Research related to improvements in these key areas has led to a better understanding of the physical phenomena that occur in Hertzian contact. Anderson and Zaretsky [Ref. 7] also state that given proper handling, installation and maintenance of lubrication, a rolling-element bearing ultimately will fail by fatigue. Since fatigue results from material weakness, much research to improve the quality of bearing materials has been accomplished.

The scope of this research is broad and considers melting techniques, component hardness combinations, material fiber orientation, ausforging, residual stresses and various aspects

of design. In their review, Anderson and Zaretsky [Ref. 7] state that one method for increasing rolling-element reliability is to eliminate or reduce the content of nonmetallic inclusions, entrapped gases and trace elements. They concluded that improved melting techniques, including vacuum induction melting (VIM) and consumable electrode vacuum arc remelting (VAR), produce a more consistent, high quality steel. In addition, these remelted steels were found to improve bearing life with each successive remelting, but that the improved bearing life did not coincide exactly with the resulting material cleanliness. The problem then becomes one of economics due to the increased cost of manufacturing metallurgically clean materials. Another aspect of processing reviewed by Anderson and Zaretsky [Ref. 7] is ausforging. It was found that it too increased the fatigue life of bearings and by the mechanism of strain-induced precipitation which results in smaller, more uniformly dispersed carbides. They also remark in closing that, while tremendous progress has been made toward extending rolling-element bearing life, gaps in bearing technology do exist and research to close these gaps is required.

Kar, Horn, and Zackay [Ref. 8] state that the area of isothermal transformation appears very attractive because it offers the potential of achieving optimum strength-toughness combinations through microstructural control. They also state that bainitic structures are important because, through choice

of proper isothermal temperatures, it is possible to get a product of deformed ferrite and carbide, which closely resembles the structure of tempered martensite and that a duplex structure of martensite and bainite can exhibit excellent mechanical properties in certain alloy steels.

Hollux [Ref. 9] has indicated that heavy duty bearings with bainitic micro-structures are presently being manufactured. Data to date indicate that the service life of these bearings is greatly extended when compared to bearings with martensitic microstructures particularly for large rolling-element bearings such as those utilized in earth moving equipment.

B. ISOTHERMAL TRANSFORMATION

The transformation of austenite at constant temperature was first investigated by Bain and Davenport in 1930 [Ref. 10]. Figure 1 is the isothermal-transformation (I-T) diagram for AISI 52100 steel austenitized at 850°C and possessing an ASTM grain size of 9. This diagram is best understood by explaining the procedure used in its determination. First, several small specimens are austenitized at a suitable temperature and are then quickly transferred to a bath held at a desired transformation temperature below A_s . Their small size allows them to cool quickly to the reaction temperature. The specimens are then held for a time to allow the transformation reaction to proceed. When the desired time has passed a specimen is

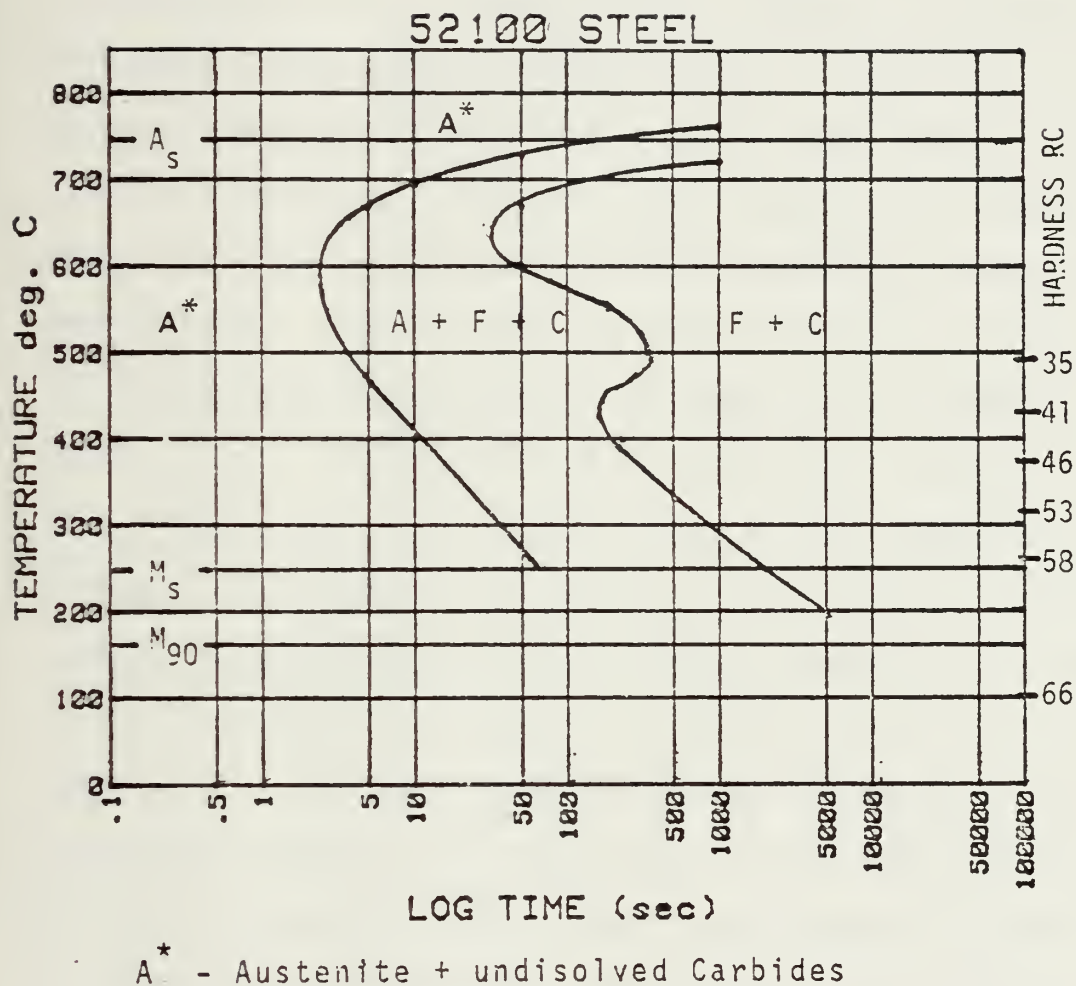


Figure 1. Isothermal transformation diagram for 52100 steel. Applicable for austenitizing at 850°C and for an ASTM grain size of 9. Adapted from Reference 11.

quenched into water or oil maintained at a temperature below M_s . This quench stops the transformation and causes any remaining austenite to change to martensite. When the specimen is examined under the microscope, it is usually easy to distinguish the portion of the austenite that reacted isothermally from the portion that changed to martensite on quenching. The data on times of reaction and the corresponding amounts of transformation products can then be plotted to give an isothermal reaction curve. Thus the I-T diagram summarizes the isothermal reaction curves for all reaction temperatures. The diagram is read from left to right and the "C" shaped curves represent the times for the beginning and completion of the transformation reaction. As the transformation temperature is decreased below A_s the isothermal reaction becomes increasingly fast because the austenite is increasingly unstable. However, at still lower temperatures the decrease in the diffusion rate becomes predominate and the transformation slows.

The properties and characteristics of the transformation products also depend upon the transformation temperature. Pearlite forms in the range above the nose of the C-curves but with decreasing temperature the lamellar spacing decreases with a resultant increase in strength and hardness. Below the nose, bainite forms with morphology changing from feathery at relatively high temperatures to platelike at lower temperatures. Since the transformation products differ in hardness as well as microstructure, additional data involving

the progress of the transformation reaction can be obtained from hardness as a function of transformation time. If the austenitized specimens are held at a transformation temperature below A_s for very short times and then quenched, a fully martensitic transformation product with high hardness will be obtained. As the holding times are increased more of the austenite will transform to either pearlite or bainite and the quenched specimen will exhibit lower hardness characteristic of the mixed martensitic, bainite or pearlite microstructure.

The object of this research is to investigate the additional effects of grain and carbide refinement on the isothermal transformation characteristics of AISI 52100 steel. It is known [Ref. 12] that grain size has a marked effect on the hardenability of steel. This is related to the I-T diagram since a steel with high hardenability is one in which austenite is able to transform to martensite without forming pearlite, even when the rate of cooling is slow. Pearlite nucleates at grain boundaries and the total number of nuclei that form per unit time varies with the amount of grain boundary area available. Since in a fine grained steel there is more grain boundary area when compared to a coarse grained material, the formation of pearlite is more rapid. Fine grain size then affects hardenability by shifting the curves of the I-T diagram to the left. Barnett and Troiano [Ref. 13] state that a fine grain size also lowers the martensite start

temperature. Although the steels they studies were of a lower carbon content than 52100 the effect may be comparable.

On the other hand, carbide refinement may also have an effect on hardenability. This may be attributed to the fine carbides dissolving more quickly during austenitization resulting in a higher carbon content in the austenite. Increased carbon content is known [Ref. 14] to increase the hardenability of steel, i.e., to shift the curves of the I-T diagram to the right. Additionally, both the martensite start and finish temperatures are reduced with increasing carbon content of the austenite.

In summary, the effect of grain and carbide refinement will be a balance of effects tending to accelerate the decomposition of austenite (grain refinement) and the effects tending to retard the decomposition of austenite (carbide refinement). In addition, these two factors will also influence the martensite start temperature, M_s . It is the objective of this research to evaluate experimentally these areas.

II. EXPERIMENTAL

A. MATERIALS AND PROCESSING

The material used in the conduct of this research was vacuum-induction melted (VIM) and consumable-electrode vacuum-arc remelted (VAR) AISI 52100 steel. It was obtained from Carpenter Steel in the form of 8.9 cm. (3.5 in.) diameter round bar which had been hot rolled to size and spheroidize annealed. Chemical analysis, by Anamet Laboratories, Inc., Berkeley, California, of this material is presented in Table I. It should be noted that this analysis shows a 0.17 weight percent tungsten content which is significantly greater than that found in the 52100 steel previously used by Chung and McCauley [Refs. 1, 2]. A 25.4 cm. (10 in.) length was sectioned from this bar, heat treated, and rolled as shown in Figure 2. The two-cycle austenitizing treatment and oil quenching was completed by Schultz [Ref. 3] and the final reheating and warm rolling was done at Viking Metallurgical, Inc., of Richmond, California. This process was one of several investigated by Schultz [Ref. 3] and was designated by him as HT-4. In subsequent work with this HT-4 material, Schultz [Ref. 3] designated it as material "D" to distinguish it from other processes. As only the one HT-4 process was used as a source of rolled material in this research, the HT-4 designation will be used here.

TABLE I

ALLOY CHEMISTRY IN WEIGHT PERCENTAGE

C	Mn	Cr	Ni	Mo	Si	S	P	Al
1.06	0.36	1.37	0.07	0.04	0.27	0.010	0.008	0.03
	Cb	Cu	Fe	Pb	Ti	W	V	
	0.005	0.03	Balance	0.005	0.006	0.17	0.02	

HT-4

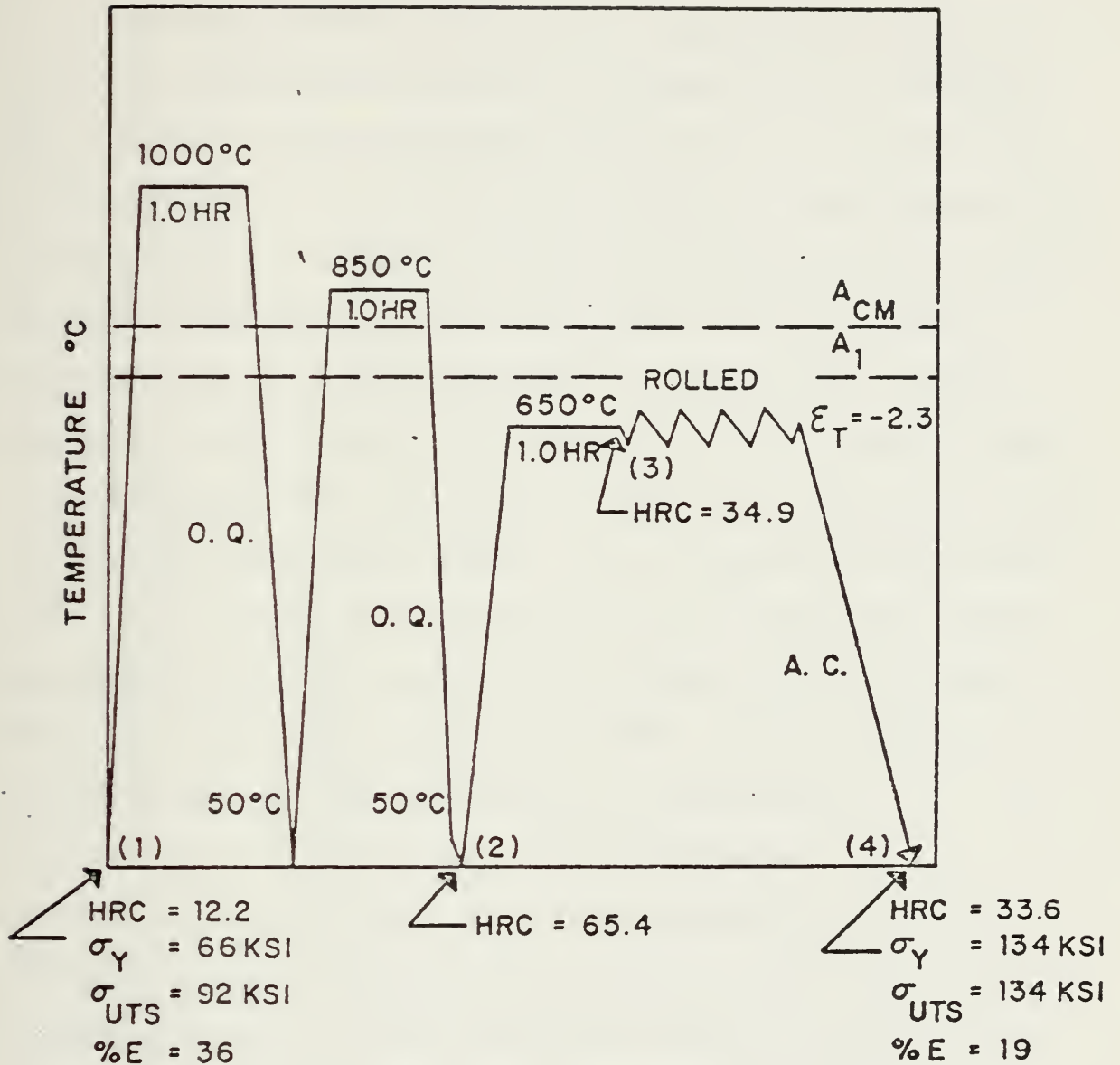


Figure 2. Schematic of thermomechanical treatment HT-4

B. ISOTHERMAL TRANSFORMATION HEAT TREATMENT

Specimens were prepared in the form of hardness coupons from both the original as-received, spheroidize annealed (AR) and the thermomechanically processed (HT-4) materials. The nominal coupon dimensions were 2.54 cm. by 1.27 cm. by 0.635 cm. (1.0 in. by 0.5 in. by 0.25 in.). The coupons were austenitized at 850°C for a time corresponding to one hour per inch of thickness. For this sample size, all were austenitized for fifteen minutes. A Thermolyne furnace, equipped with a Dubeque III solid state controller was used and temperature was maintained to within ± 5 degrees Celsius. The coupons were then removed from the furnace and quenched into neutral salt in a Lindberg type CR-5 salt bath furnace and held for times ranging from 5 seconds to 100 minutes. The salt bath was maintained at 350°C, 275°C or 250°C, representing the three isothermal transformation temperatures investigated. The temperatures of both the furnace and the salt bath were constantly monitored throughout the treatments using a Newport Electronic Digital Thermometer with a stainless-steel sheathed chromel-alumel thermocouple positioned at the specimen location on the hearth or in the bath. As each specimen reached the desired point in time of transformation it was removed from the neutral salt bath and oil quenched to room temperature, 20°C.

C. HARDNESS TESTING

To remove any surface scale obtained during heat treatment each specimen was ground through 400 grit paper. Twelve initial Rockwell-C hardness measurements were then taken using a Wilson Model 1-JR Rockwell Harness Tester. Results were determined by discarding the high and low values and the statistical mean and standard deviation of the ten remaining values were then calculated using an internal function of a TI-59 calculator. Since the Thermolyne furnace utilized for austenitization did not provide decarburization protection, the specimens were again ground through 400 grit to remove the initial hardness test indentations. Twelve hardness values were again taken and results obtained in the manner previously described. These hardness values were recorded for the temperatures for each transformation time. Specimens were then selected at the beginning, midpoint and end of the transformation stage for each temperature and sectioned in half, with one half set aside for microstructural examination. The remaining portion was again ground through 400 grit to remove any previous hardness test indentations and then immersed in liquid nitrogen ($T = -196^{\circ}\text{C}$) for one hour. Eight Rockwell-C hardness readings were then obtained and the statistical mean and standard deviation calculated after discarding the high and low values.

D. OPTICAL MICROSCOPY

Specimens previously selected, sectioned, and set aside for examination were mounted in bakelite. A Buehler Automet grinding and polishing unit was utilized to grind the specimens through 600 grit paper and then to polish them with 1.0 micron Alpha Alumina and 0.5 micron Gamma Alumina micropolish in successive steps. After polishing, the specimens were washed in an ethanol bath and etched in a 5% nital solution for times variously from 5 seconds to 1 minute. A Zeiss Universal Photomicroscope was then used to perform optical photomicroscopy of each specimen.

E. TRANSMISSION ELECTRON MICROSCOPY

Transmission electron microscopy was performed on carbide extraction replicas at Stanford University's Hopkins Marine Station using a Philips EM201 transmission electron microscope. Specimens mounted for optical microscopy were also used for the carbide extraction replication. To avoid pitting, each specimen was metallographically prepared using a Buehler Automet grinding and polishing unit with polishing cloth and 1.0 micron diamond paste. After polishing, the specimens were washed in an ethanol bath and lightly etched in a 2.5% nital solution. Typical etching times were approximately five seconds. A Fullam Model 1250 Evaporator with a vacuum of approximately 6×10^{-5} torr was used to evaporate carbon onto the etched specimen surface. A burn time of approximately

three seconds with a filament current of 50 amps was used. The coated surface was then sectioned into a 2 mm. grid pattern using a razor knife. The specimen was then immersed in a 15% nital solution until the replica began to lift. The replica was then floated off the specimen surface in an aqueous 3% ethanol solution, retrieved on a standard 3 mm. copper specimen grid, and allowed to dry prior to microscopic examination.

F. RETAINED AUSTENITE BY X-RAY DIFFRACTION

Specimens previously mounted for optical microscopy were utilized for x-ray determination of retained austenite. This analysis was accomplished using a Picker X-ray Diffractometer located at Lawrence Berkeley Laboratories, University of California, Berkeley, California. Lithium-fluoride monochromated copper radiation was employed and diffraction peaks between 60° and 95° in 2θ were autographically recorded. Peaks of interest were the $(220)_{\gamma}$, $(211)_{\alpha}$, and $(311)_{\gamma}$. Retained austenite was evaluated by measuring the integrated intensity and the volume fraction of retained austenite was determined using the relation given by Miller [Ref. 15]:

$$I_{\gamma} = \frac{I_{(220)\gamma} + I_{(311)\gamma}}{2} \quad \text{eqn. 1,}$$

and

$$V_{\gamma} = \frac{1.25I_{\gamma}}{I_{\alpha} + 1.25I_{\gamma}} \quad \text{eqn. 2,}$$

where I_{γ} = average gamma integrated intensity,
 $I_{(220)\gamma}$ = the integrated intensity of the $(220)\gamma$
peak, $I_{(311)\gamma}$ = the integrated intensity of the
 $(311)\gamma$ peak, I_{α} = the integrated intensity of the
 $(211)\alpha$ peak, and V_{γ} = the volume fraction of the
retained austenite.

G. TENSILE TESTING

Rectangular subsize tensile specimens were machined according to ATSM specifications [Ref. 16] prior to isothermal heat treatment. Specimens were machined from both the original spheroidize annealed (AR) and thermomechanically processed (HT-4) materials. The tensile specimens were then isothermal heat treated using the equipment and method described in section B. Isothermal transformation heat treatments at 350°C and 275°C were accomplished for 20 and 50 minutes, respectively. The 250°C heat treatment utilized in other sections was eliminated due to the high hardness of the material after transformation and the inability of available test equipment to grip specimens of this high hardness. Tensile tests were conducted on an Instron Model TT-D Universal Testing Instrument utilizing wedge action grips to minimize non axial loading of the specimens. Load-versus-elongation curves were autographically recorded while the specimens were loaded to failure at an extension rate of 5.1 mm. (0.2 in.) per minute. The data obtained from these

tests were subsequently converted to engineering stress versus engineering strain to determine the ultimate tensile strength. Standard methods were also employed to calculate the percent elongation and percent reduction in area to failure.

H. DIFFERENTIAL THERMAL ANALYSIS (DTA)

The DTA data was obtained at Defense Systems Division, Honeywell Inc., Hopkins, Minnesota. Cylindrical samples 3.17 mm. (0.125 in.) long and 3.17 mm. (0.125 in.) in diameter were machined from both the AR and HT-4 materials. DTA was performed with a heating rate of 10 degrees celsius per minute and the data provided in the form of autographic recordings of the power required to maintain the heating rate versus the temperature. Using this method, both endothermic and exothermic peaks were obtained.

III. RESULTS AND DISCUSSION

A. INITIAL MICROSTRUCTURES

The microstructures of the original spheroidize annealed (AR) and thermomechanically processed (HT-4) materials were examined prior to isothermal transformation heat treatment. The microstructure of the AR material (Fig. 3a) consists of spheroidal carbide particles in a ferrite matrix. The ferrite grain size is 16-20 $\mu\text{m.}$, while the spheroidal carbides appear to be about 2-3 $\mu\text{m.}$ in size. The microstructure of the thermomechanically process material is shown in Figure 3b. The apparent fineness of this as-rolled microstructure prevents detailed optical microscopy, however this structure contains some optically resolvable carbides which will subsequently be referred to as residual carbides. They are carbides not dissolved in the thermomechanical processing [Ref. 17].

Carbide extraction transmission electron micrographs for these same AR and HT-4 materials are shown in Figures 4a and 4b. Figure 4a clearly shows the size and distribution of the carbides present in the original spheroidize annealed material. Figure 4b is a carbide extraction micrograph of the thermomechanically processed HT-4 material. Here the carbide structure is resolved; the residual carbides are about 0.5 $\mu\text{m.}$ in size and the carbides precipitated and refined during the warm rolling are 0-1 to 0.2 $\mu\text{m.}$ in size. Comparison of this

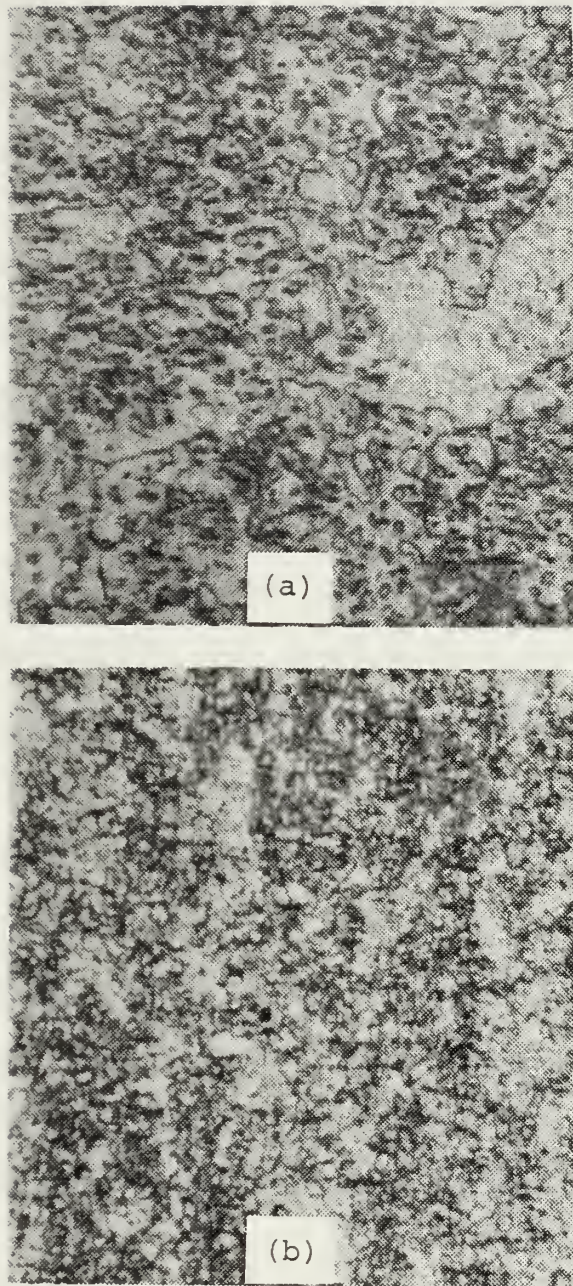


Figure 3. Optical micrographs of AISI 52100 steel. (a) standard spheroidize annealed microstructure consisting coarse spheroidal carbides in a ferrite matrix; (b) thermomechanically refined HT-4 microstructure consisting of submicron spheroidal carbides in a fine grained ferrite. Etched in 5% nital solution. 1600X.

micrograph to that of Figure 4a clearly shows the extent to which this processing refines the carbide structure. The ferrite grain size for this material was determined to be 0.6-0.8 μm . by thin foil transmission electron microscopy [Ref. 17]. Figure 4c is a separate carbide extraction micrograph of the HT-4 thermomechanically processed material and is included to illustrate the difficulty that may be encountered when utilizing the carbide extraction replication method. The carbides here appear to be 3-4 μm . in diameter, in contrast to the submicron size of the carbides evident in Figure 4b. Additionally, the apparent volume fraction of carbides present in this micrograph is much greater. Quantitative analysis of this micrograph suggests the volume fraction to be nearly three times that possible for AISI 52100 steel. These results are believed to have been caused by an overly long etch in the 15% Nital solution used to lift this replica. This long etch may have resulted in a much greater volume of the specimen being subjected to the extraction process and hence the number of carbides extracted was increased.

B. ISOTHERMAL TRANSFORMATION

Specimens from both the AR and HT-4 materials were austenitized at 850°C and isothermally transformed at 350°C, 275°C or 250°C for the three isothermal transformation heat treatments investigated. Specimens were isothermally transformed

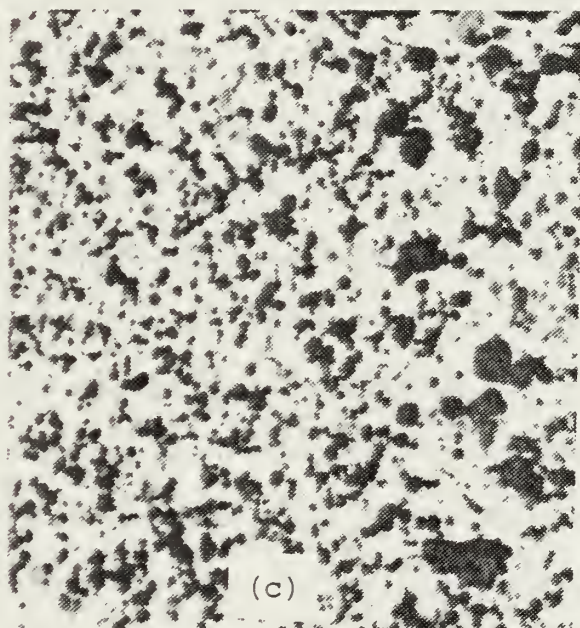
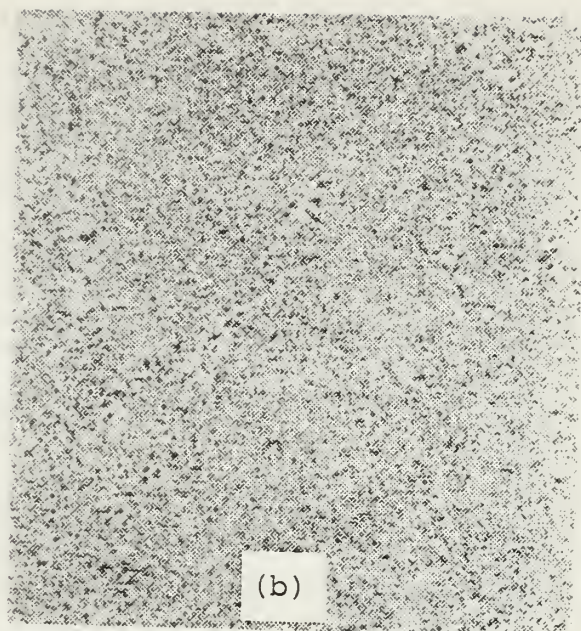


Figure 4. Carbide extraction transmission electron micrographs of AISI 52100 steel. (a) standard spheroidize annealed carbide microstructure, (b) thermomechanically refined HT-4 carbide microstructure consisting of submicron residual and tempered carbides. 1400X. (c) carbide extraction micrograph of overly extracted thermomechanically refined HT-4 material. 5000X.

for times ranging from 5 seconds to 100 minutes for each transformation temperature. At selected times of transformation, individual specimens were oil quenched to room temperature, prepared and Rockwell-C hardness values obtained. These hardness values were then plotted versus the log of transformation time and this data is shown in Figures 5 through 7.

Figure 5 is a plot of the results obtained for the 350°C isothermal transformation heat treatment. The hardness of the specimens which were transformed for very short times (5-10 seconds) is about Rc 65. This hardness is consistent with the martensitic microstructure predicted by the isothermal transformation diagram (I-T diagram), Figure 1. For longer times, the hardness values are seen to decrease and eventually reach a constant lower value. Again, this reflects the expected trend in hardness predicted by the I-T diagram since a duplex microstructure of high hardness martensite and a lower hardness bainite will occur at intermediate transformation times, giving way to a lower hardness, completely bainitic microstructure at longer times. It is also evident that the HT-4 material closely follows the transformation characteristics of the AR material.

Figures 6 and 7 show the results for the 275°C and 250°C isothermal transformation heat treatments respectively. In the comparison of Figures 5 through 7 it is seen that the time at which the hardness values first begin to decrease

ISOTHERMAL TRANSFORMATION

350 deg C

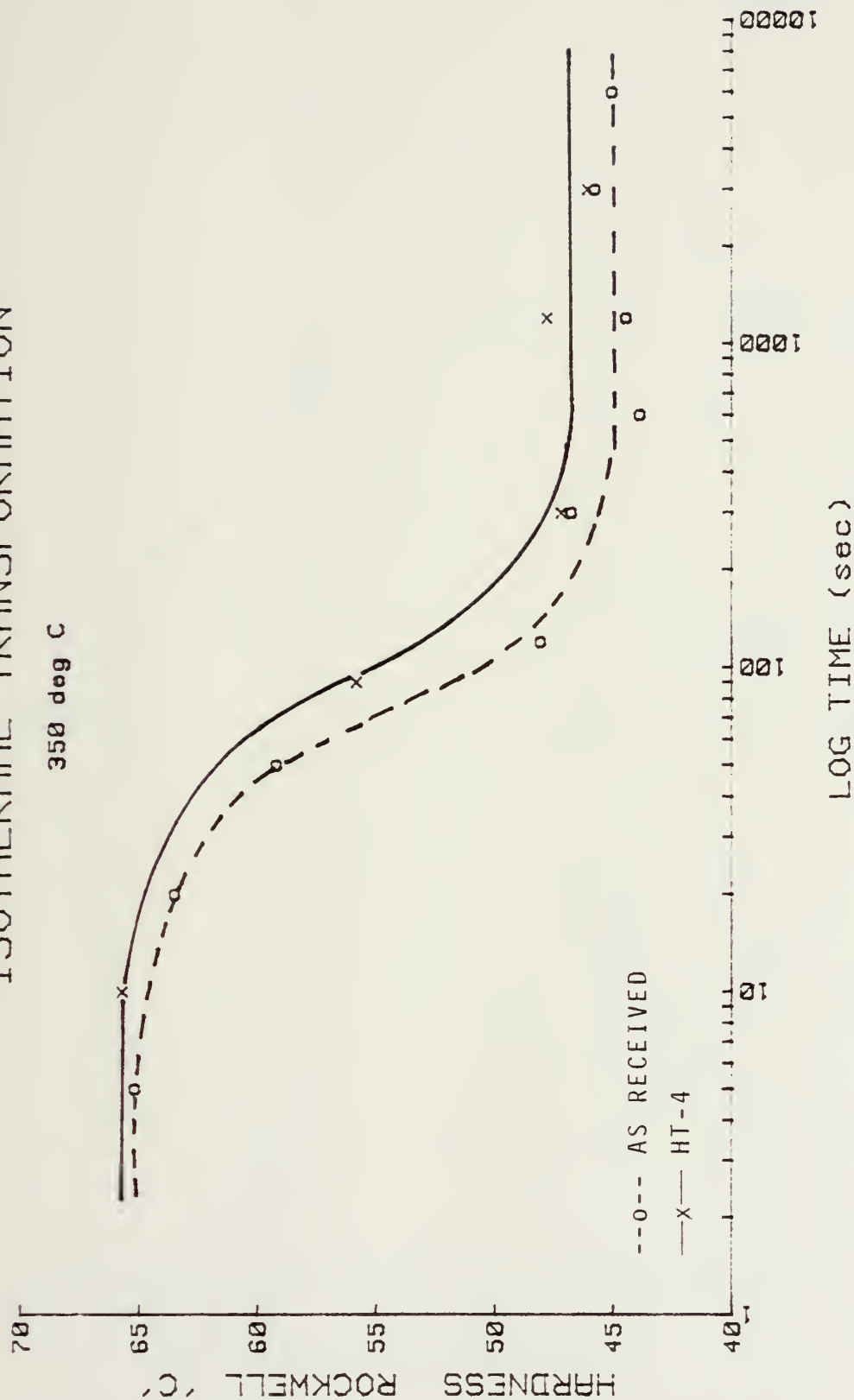


Figure 5. Plot of hardness versus time for both AR and HT-4 materials. Austenitized at 850°C and isothermal transformation heat treated at 350°C for times variously from 5 seconds to 100 minutes.

ISOTHERMAL TRANSFORMATION

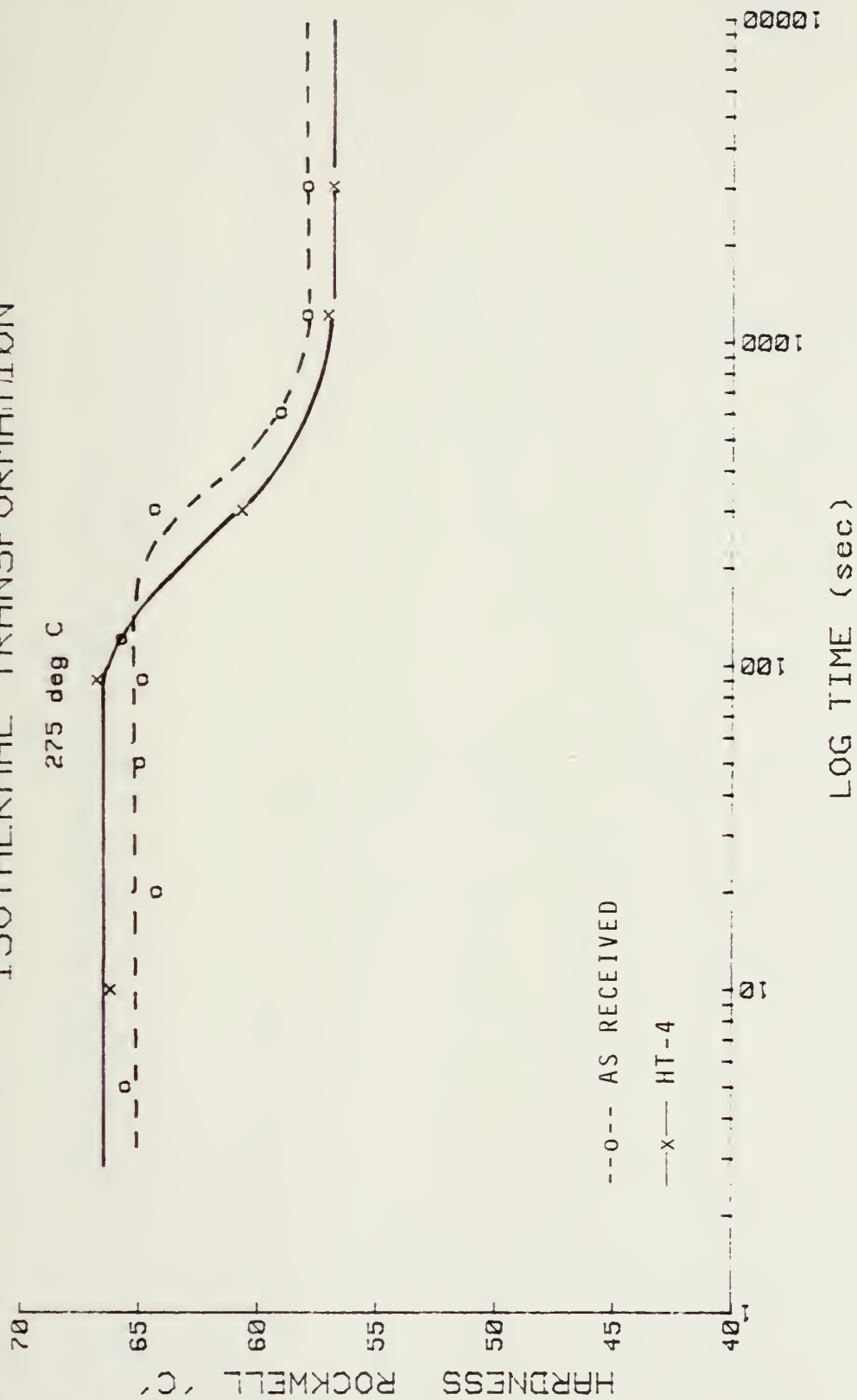


Figure 6. Plot of hardness versus time for both AR and HT-4 materials. Austenitized at 850°C and isothermal transformation heat treated at 275°C for times variously from 5 seconds to 50 minutes.

ISOTHERMAL TRANSFORMATION

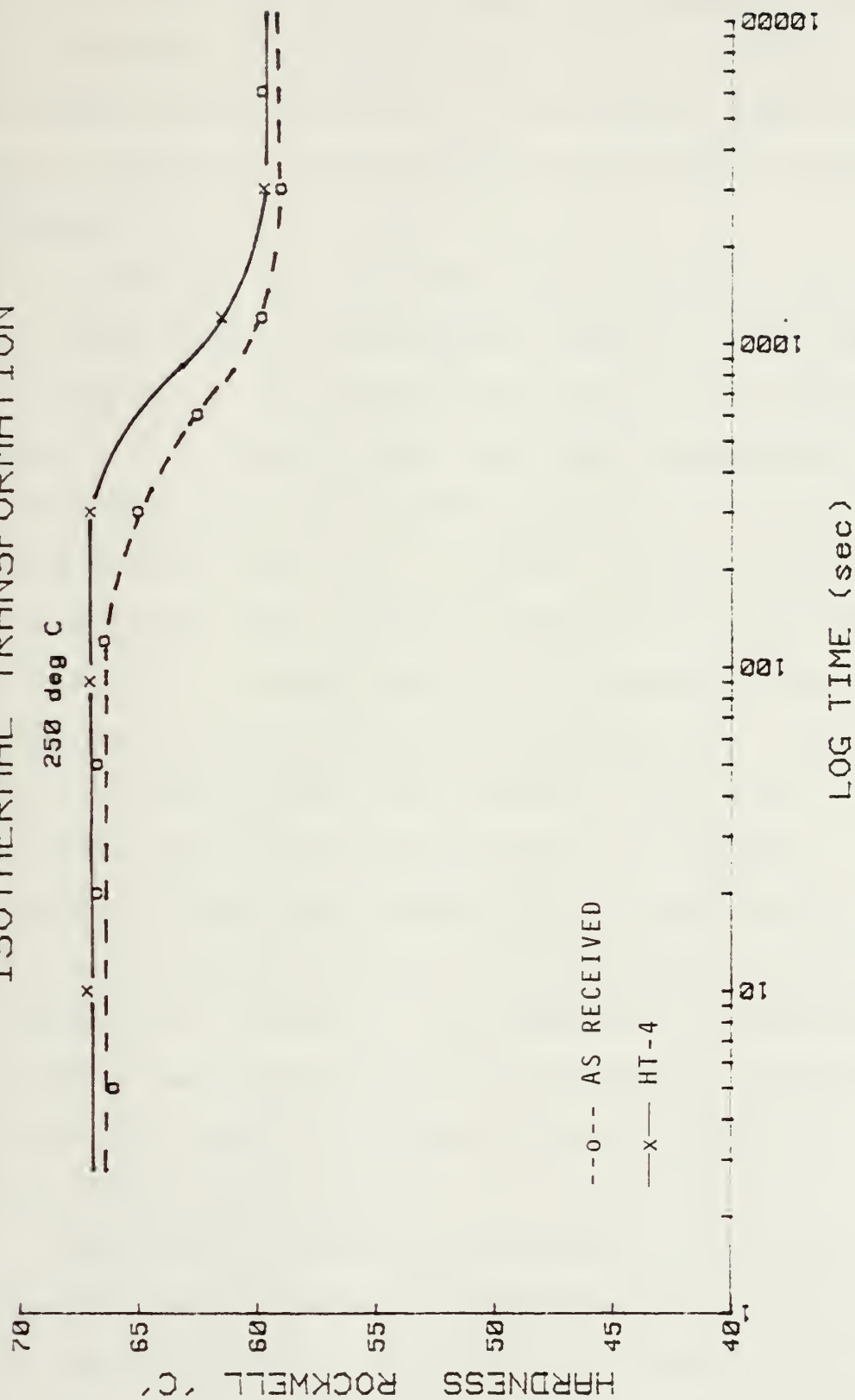


Figure 7. Plot of hardness versus time for both AR and HT-4 materials. Austenitized at 850°C and isothermal transformation heat treated at 250°C for times variously from 5 seconds to 100 minutes.

as the isothermal transformation heat treatment temperature is decreased. Additionally, the times to reach the lower hardness and the magnitude of this hardness both increase as the isothermal transformation heat treatment temperature decreases.

In Table 2, the isothermal transformation reaction start and finish times as suggested by Figures 5-7 are summarized and compared to the reaction start and finish times obtained from the I-T diagram. This table also compares the hardness of the completely transformed materials in Figures 5-7 to those obtained from the I-T diagram. As the isothermal transformation heat treatment temperature is decreased it is seen that, in both materials, the times for the transformation reaction start and finish increase. The reaction start and finish times from Figures 5-7 are also in general agreement with those predicted by the I-T diagram. This supports the view that hardness versus transformation time is a valid representation of the progress of the isothermal transformation reaction. The comparison of experimental and predicted hardness values for the completely isothermally transformed materials is also in close agreement and supports the procedures utilized.

The results presented in Figures 5-7 and in Table II show that the isothermal transformation characteristics of the thermomechanically processed HT-4 material are essentially identical to those of the AR material. As noted previously,

TABLE II

ISOTHERMAL TRANSFORMATION CHARACTERISTICS

<u>Times of Transformation in Seconds</u>									
Isothermal Transformation Temperature °C	I-T Diagram		AR		Experimental HT-4				
	Start	Finish	Start	Finish	Start	Finish			
350	20	350	9	400	15	500			
275	50	1200	150	1500	90	2000			
250	60	2000	100	2500	300	3000			

<u>Transformed Microstructure Hardness</u>									
					Rockwell-C				
					AR		HT-4		
					I-T Diagram				
					Start	Finish			
350					47	44	46		
275					56	58	57		
250					60	59	60		

fine grained steels are less hardenable than coarse grained steels. This result is usually attributed to the presence of more nucleation sites for transformation products such as pearlite or bainite and hence a leftward shift in the start and finish times on the I-T diagram. If the grain refinement attained in the HT-4 material is retained during heat treatment, then other factors must offset the effect of grain refinement. In this case possibly the refined carbides dissolve more quickly upon austenitizing, resulting in a higher carbon austenite which will tend to increase hardenability, i.e., shift the start and finish times to the right. Other factors may include the presence of carbides not dissolved during austenitizing such that they serve as nucleation sites during subsequent isothermal transformation and hence control the transformation in place of the austenite grain boundaries. This would require coarsening of the initially fine carbides in the HT-4 material. Subsequent sections of this chapter will consider each of these factors.

C. MICROSCOPIC EXAMINATION

Optical and carbide extraction replication micrographs were examined from selected times of transformation for the AR and HT-4 materials in order to address the question of the dominant mechanism for isothermal transformation. Figures 8a-c present the microstructure of the AR material at three different isothermal transformation times. Figure 8a is the microstructure after 850°C austenitization followed by a

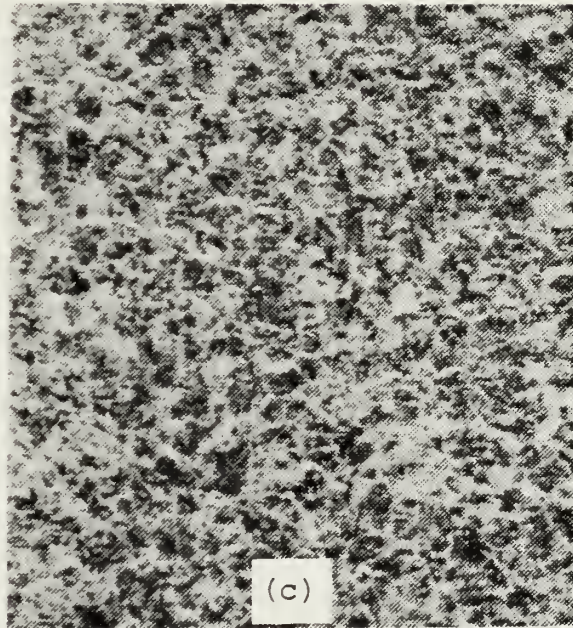
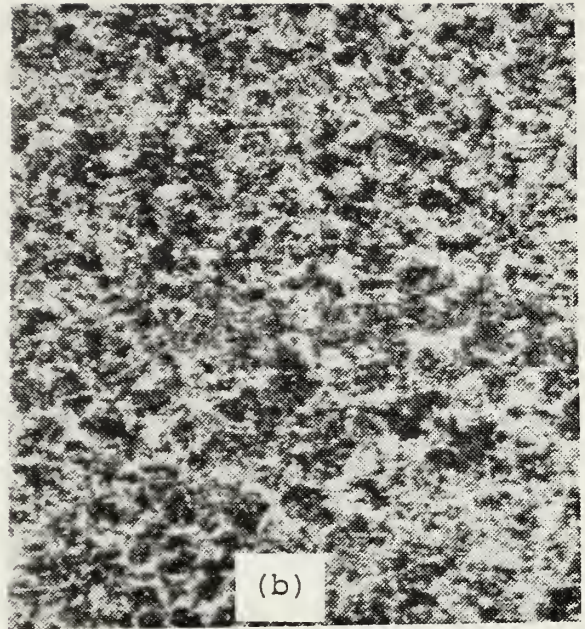
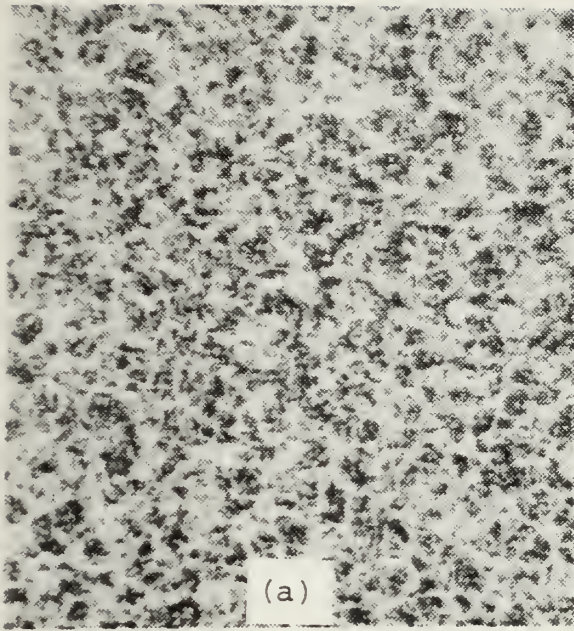


Figure 8. Optical micrographs of the AR material after 850°C austenitization and subsequent isothermal transformation heat treatment at 275°C. After: (a) 5 seconds, (b) 10 minutes, (c) 50 minutes. Micrographs reveal a grain size of 15-16 μm . and some residual carbides are resolved. Etched in 5% nital solution. 250X.

five second isothermal transformation period. In this low magnification micrograph the grain size is seen to be 15-16 μm and is comparable to that of the parent material. Residual carbides are also evident but at this low magnification a detailed analysis is not possible. Figures 8b and c illustrate the microstructures of the AR material isothermally transformed at 275°C for the mid and completion points in time for the transformation reaction. Comparison of these micrographs with Figure 8a indicates that the grain size, number and size of residual carbides remains constant.

Figures 9a through c are micrographs of the thermomechanically refined HT-4 material for similar transformation times at 275°C . The microstructure in Figure 9a is of this refined material after an 850°C austenitization and 275°C isothermal transformation for 10 seconds. This micrograph as well as those in Figure 9b and c show that the relative fineness of the microstructure in the thermomechanically processed material is retained. The fineness of the microstructure in these micrographs prohibits a detailed analysis but the degree of grain refinement and the apparent lack of optically resolvable residual carbides is clear. Close examination also reveals that grain coarsening has taken place but they remain approximately 3-4 μm in size.

Higher magnification optical micrographs corresponding to those of Figures 8 and 9 are shown in Figures 10a-c for the AR material, Figure 11a-c for the HT-4 thermomechanically

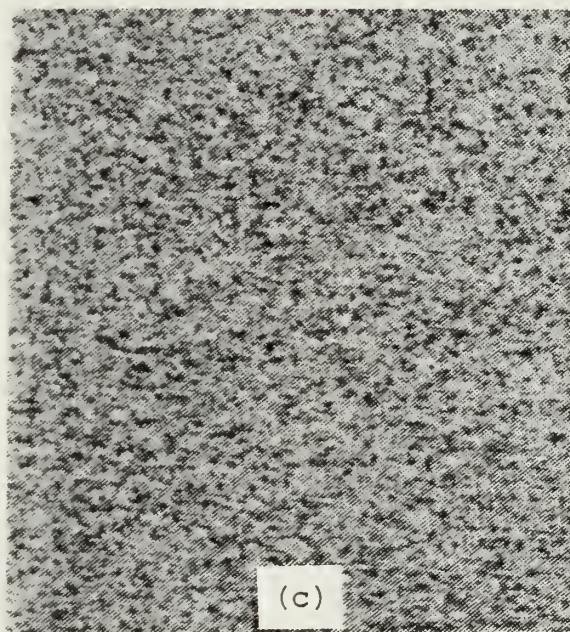
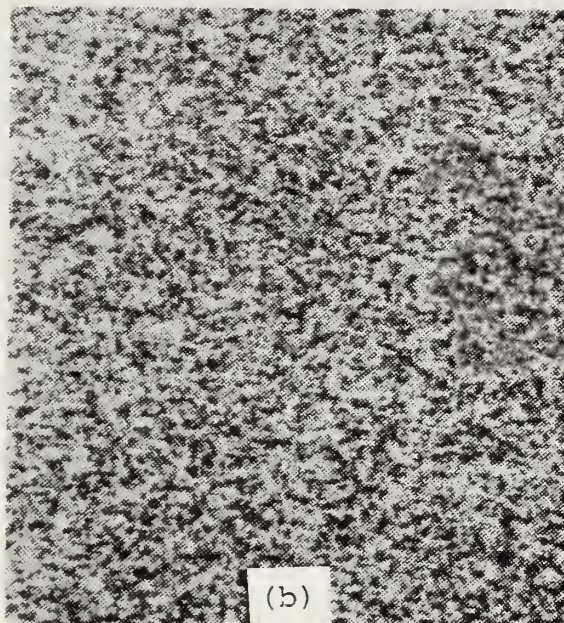
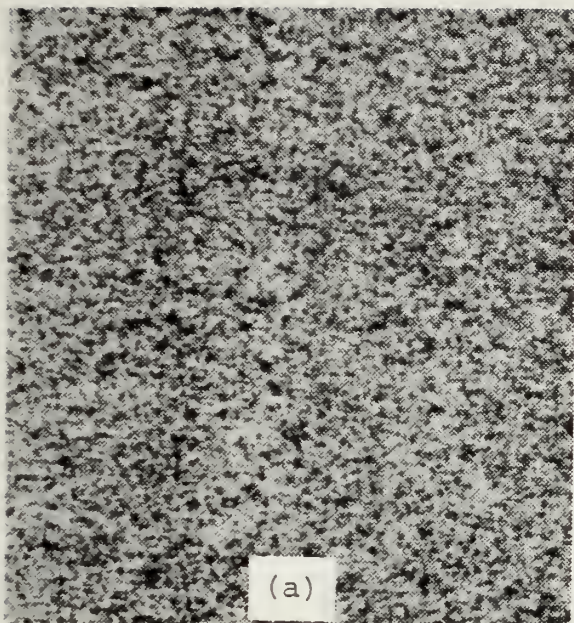


Figure 9. Optical micrographs of the HT-4 material after 850°C austenitization and subsequent isothermal transformation heat treatment at 275°C. After: (a) 10 seconds, (b) 5 minutes, and (c) 50 minutes. Micrographs indicated a slight coarsening of the grains. The grain size here is 3-4 μm. Etched in 5% nital solution. 250X.

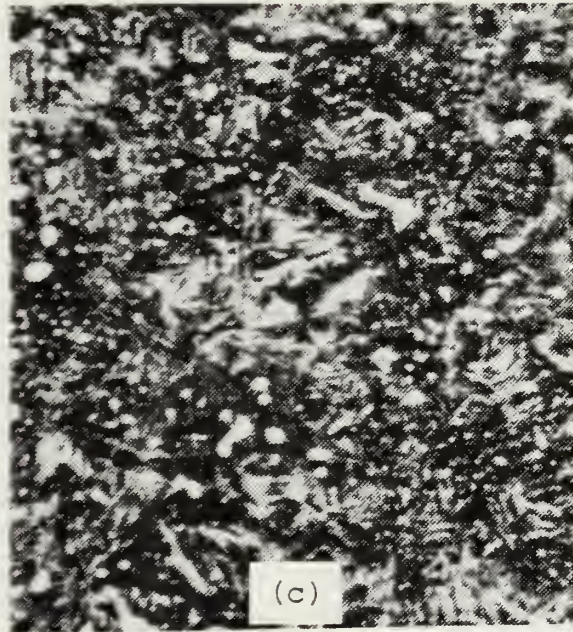
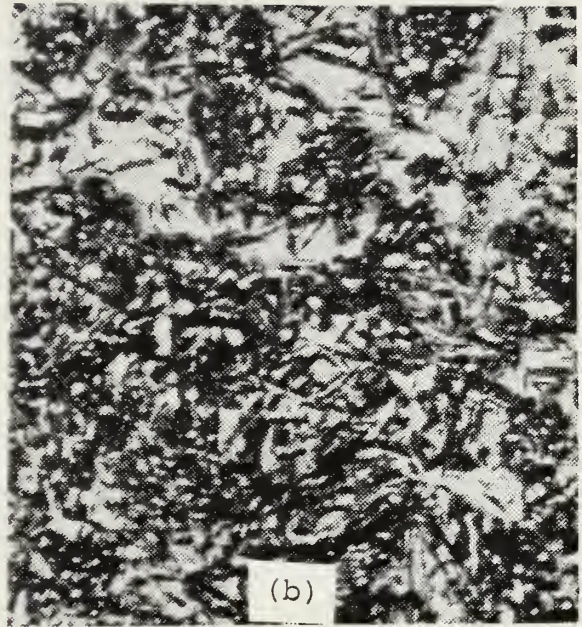
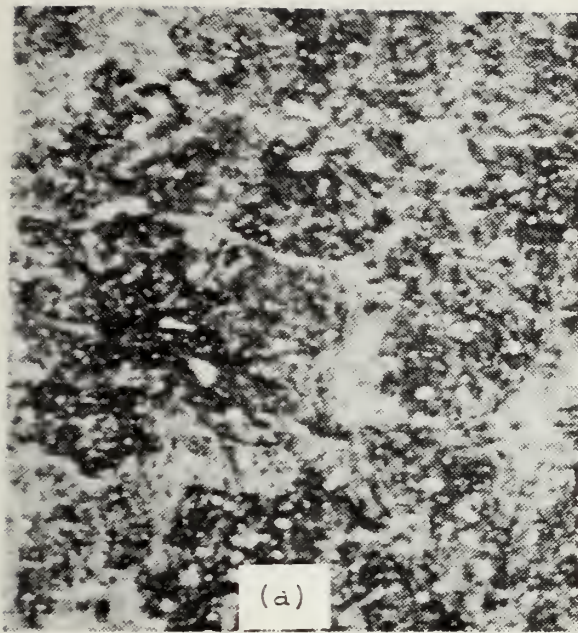


Figure 10. Optical micrographs of the AR material after 850°C austenitization and subsequent isothermal transformation heat treatment at 275°C. After (a) 5 seconds, (b) 10 minutes, and (c) 50 minutes. Also the microstructures are (a) martensite, (b) duplex martensite and bainite, and (c) bainite. Residual carbides are evident throughout. Etched in 5% nital. 1600X.

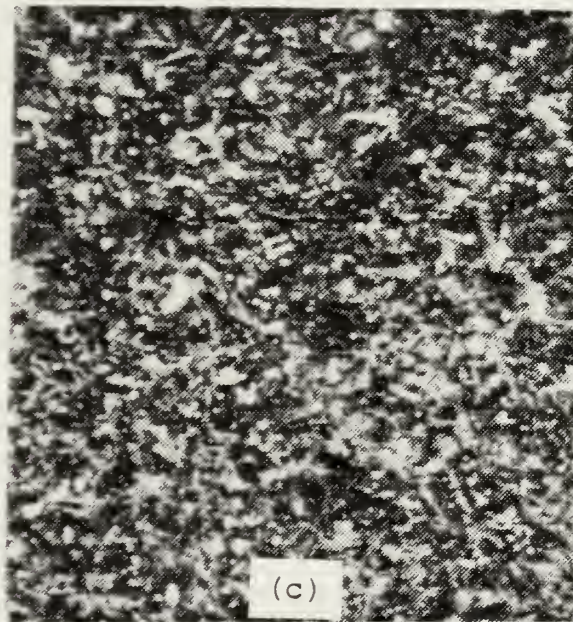
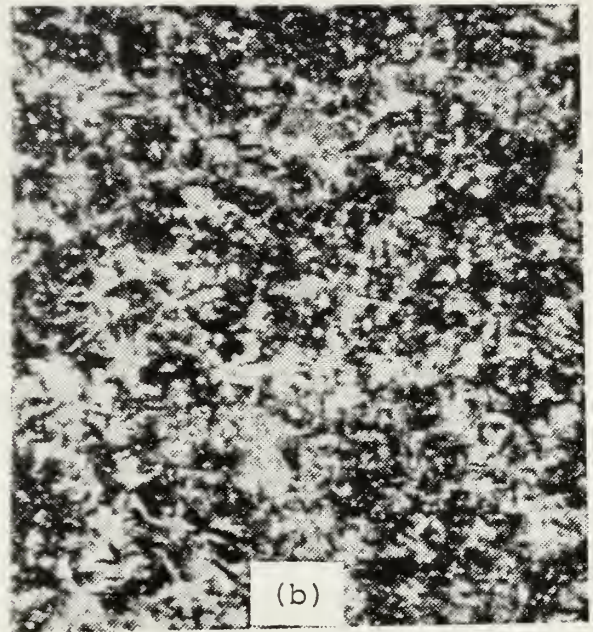
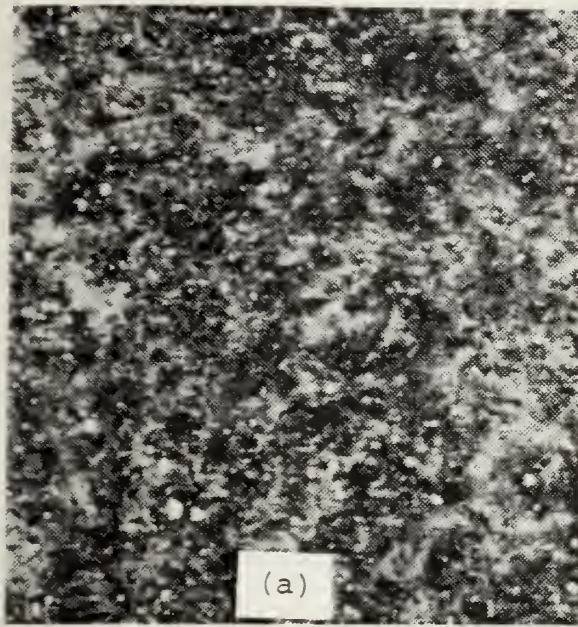


Figure 11. Optical micrographs of the HT-4 material after 850°C austenitization and subsequent isothermal transformation heat treatment at 275°C. After (a) 10 seconds, (b) 5 minutes, and (c) 50 minutes. The microstructures are: (a) martensite, (b) duplex martensite and bainite, and (c) bainite. The carbides coarsened during austenitization are evident. Etched in 5% nital solution. 1600X.

processed material. These micrographs more clearly reveal grain size and the residual carbide size and distribution. Examination of these micrographs again indicate no measurable growth in grain or carbide size for the AR material but do demonstrate a significant increase in the residual carbide size for the HT-4 material when compared to the parent, thermomechanically refined microstructure. This result is different from that obtained by Chung [Ref. 1] and McCauley [Ref. 2]. Both investigated various heat treatments in their work and observed little or no carbide coarsening in thermomechanically processed material. In contrast, Schultz [Ref. 3] did report such coarsening. The material used here is the same as that used by Schultz [Ref. 3], while material from different heats of 52100 steel was used by Chung [Ref. 1] and McCauley [Ref. 2]. The alloy chemistry data for the steel of this investigation reveals the presence of 0.17 wt. pct. Tungsten (W). The previously examined materials contained much less W, typically 0.02 wt. pct.; otherwise, the various materials are essentially identical. The effect of W addition to 52100 steel is not known although W in general closes down the austenite phase field [Ref. 18] and raises the solvus temperature for carbon in austenite. In addition, W is a strong carbide former. It is difficult at this point unambiguously to identify the reason for this apparent difference in response of the carbides, however the presence of the W in this alloy may be very significant.

Figure 10a is the microstructure of the AR material after austenitization and isothermal transformation at 275°C for 5 seconds followed by oil quenching to room temperature. The microstructure is martensite with residual carbides clearly evident. Micrographs of the material after transformation to the mid and complete points in time of transformation are presented in Figures 10b and c. Figure 10b shows a duplex martensite and bainite microstructure and Figure 10c is that of the fully bainitic microstructure of the completely transformed AR material. Throughout this series of micrographs the relatively coarse residual carbides of the AR isothermal transformation heat treated material are clearly seen.

Micrographs of the HT-4 material for the same 275°C isothermal transformation heat treatment are presented in Figures 11a-c. Figure 11a is the microstructure after 10 seconds of isothermal treatment and is martensite with residual carbides present. Here it can be seen that the residual carbides have coarsened and many now appear to be nearly as large as those present in the heat treated AR material. The micrographs in Figures 10b and c show the effect of increased time of isothermal transformation. Figure 10b depicts a duplex martensite and bainite while Figure 10c is the microstructure of the fully transformed material and is bainite. These micrographs again reveal the size and distribution of the residual carbides and it

is seen that the carbides do not undergo any significant change during the isothermal transformation heat treatment.

To gain a better understanding of the residual carbides present in these isothermal transformation heat treated materials, carbide extraction transmission electron microscopy was performed. Figures 12a-d are carbon extraction micrographs of the AR and HT-4 materials after an 850°C austenitization and subsequent 275°C isothermal transformation heat treatment. Extraction replica micrographs of the AR material after 5 seconds and 50 minutes at the isothermal transformation temperature are presented in Figures 12a and b. Figures 12c and d are of the HT-4 thermomechanically refined material isothermally heat treated at the same temperature for 10 seconds and 50 minutes. These carbide extraction micrographs clearly show the size and distribution of the residual carbides within the microstructure. The large degree of carbide coarsening during austenitization of the HT-4 material is depicted in Figure 12c and a comparison of this to Figure 12d reveals that significant further coarsening does not occur during isothermal transformation. Other than the residual carbides, the microstructures of Figures 12a and c are martensitic while Figures 12b and d show carbon extractions obtained from a bainite microstructure. The carbides from the bainite appear as the darker, feathery regions in the micrograph. Quantative analysis of the residual carbides in Figures 12b and d determined that the

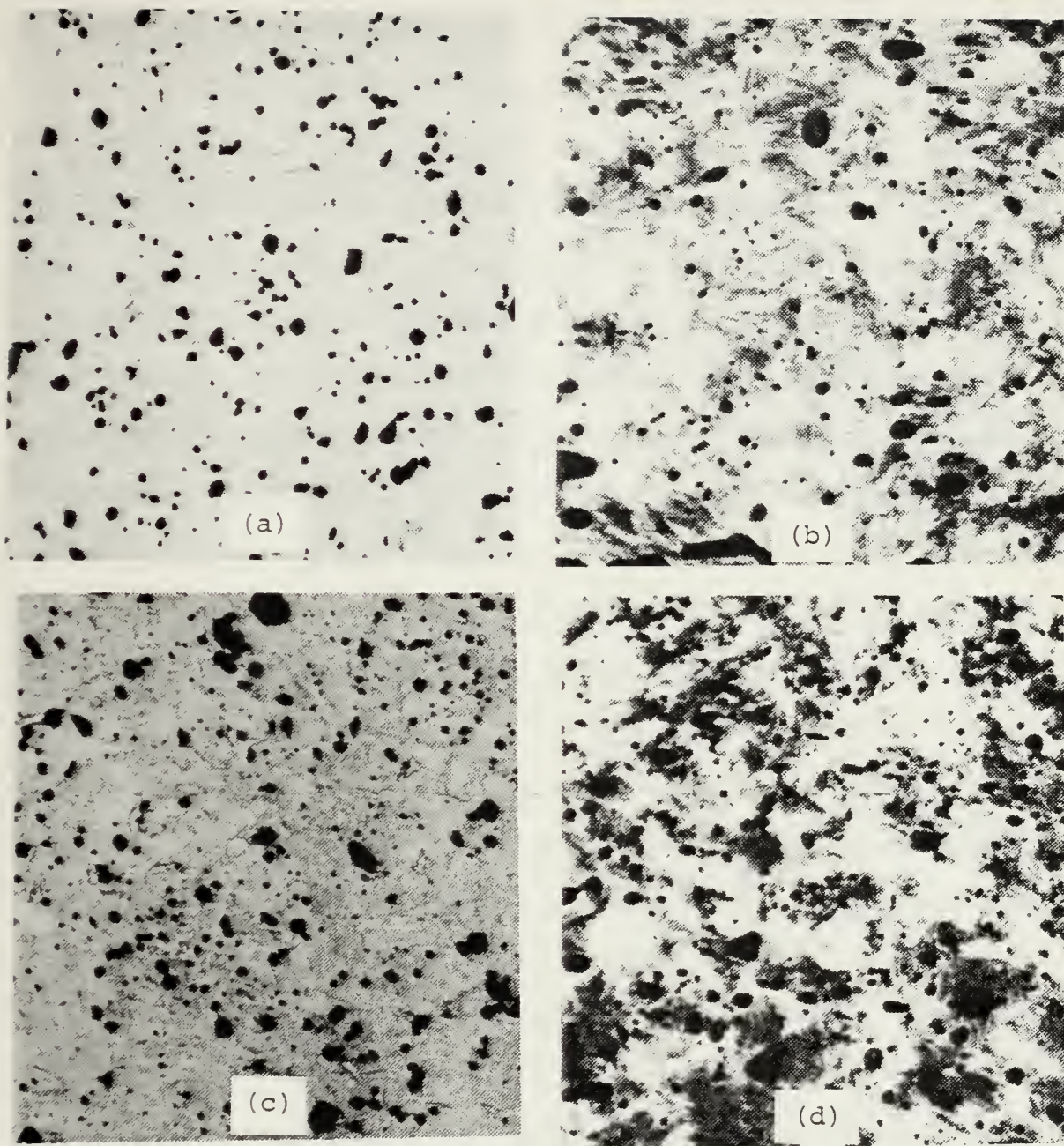


Figure 12. Carbide extraction replica micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 275°C. (a) AR after 5 seconds, (b) AR after 50 minutes, (c) HT-4 after 10 seconds, and (d) HT-4 after 50 minutes. The micrographs show the coarsening of the carbides after austenitization of the HT-4 material. 5000X.

volume percent carbide to be $7.1 \pm 0.5\%$ in the HT-4 material, and $6.5 \pm 0.5\%$ in the AR material. These values are in close agreement with those reported by Santiago [Ref. 19] for spheroidize annealed AISI 52100 steel austenitized at 845°C and indicate a matrix carbon content of approximately 0.63%.

Thus far the significant findings are that the grain size of the thermomechanically refined HT-4 material is retained throughout austenitization at 850°C and throughout subsequent isothermal transformation heat treatment. A slight coarsening does appear to take place during this heat treatment but the grain size is still significantly more refined than that of the AR material. However, this retention of refinement is not seen in the residual carbides present. The fine, submicron residual carbides present in the HT-4 refined material prior to isothermal transformation heat treatment coarsen during austenitization and the carbon content of the matrix becomes the same as that in the AR material. If the carbon content of the matrix is the same for both materials yet the grain size of the HT-4 thermomechanically processed and austenitized material is finer it would be expected that the isothermal transformation reaction kinetics for the materials would differ. Since the residual carbides in these austenitized and isothermal transformation heat treated materials are the same size it may be possible that these residual carbides have a predominant effect upon the isothermal transformation reaction kinetics.

If this is so the results of the hardness versus time of transformation investigation are supported and found to be consistant.

The microstructures for the 350°C isothermal transformation heat treatment are shown in Figures 13 and 14. Figures 13a and 14a depict the microstructure of the as received material, austenitized and isothermally transformed for 20 seconds. The microstructure is martensite with residual carbides present. Figures 13b and 14b show the microstructure after 20 minutes of isothermal transformation to be bainite with residual carbides. Micrographs of the HT-4 material at the same points in the isothermal transformation heat treatment are presented in Figures 13c and 14c and d. The microstructure is again martensite at the short times of transformation and bainite at longer times with residual carbides again present in all micrographs. A comparison of these micrographs reveals that the results of the 350°C isothermal transformation heat treatment are very similar to those obtained from the 275°C heat treatment. The grain size of the thermomechanically refined material is retained throughout the heat treatment while the carbides in this refined material coarsen during austenitization and appear to be comparable in size to those in the AR material.

Figures 15 and 16 are micrographs comparing the HT-4 and AR materials after isothermal transformation heat treatment at 250°C at short and long times of isothermal transformation. Comparison of these micrographs again reveal

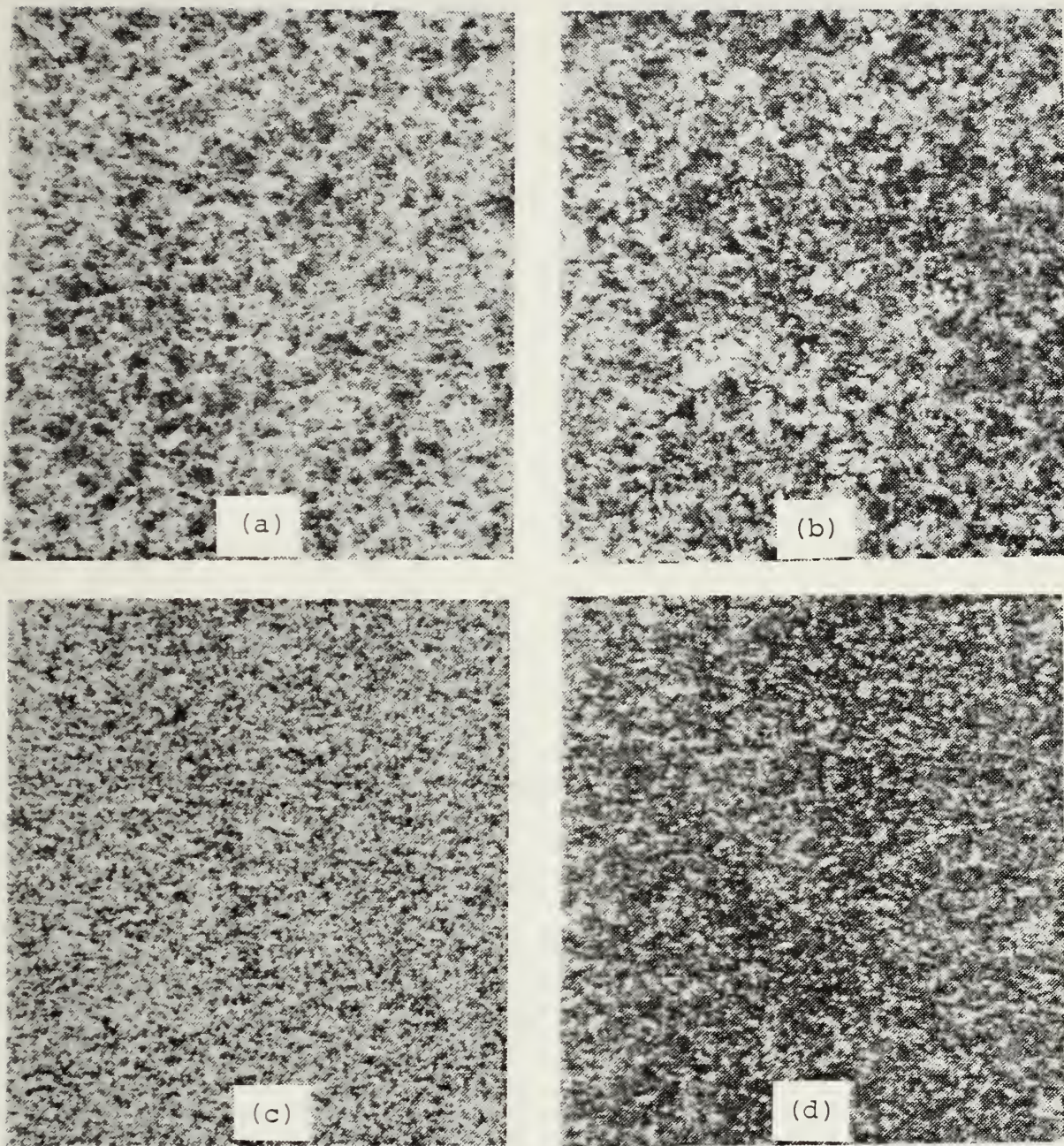


Figure 13. Optical micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 350°C. (a) AR after 20 seconds, (b) AR after 20 minutes, (c) HT-4 after 10 seconds, and (d) HT-4 after 20 minutes. Microstructures are (a & c) martensite and (b & d) bainite. Etched in 5% nital solution. 250X.

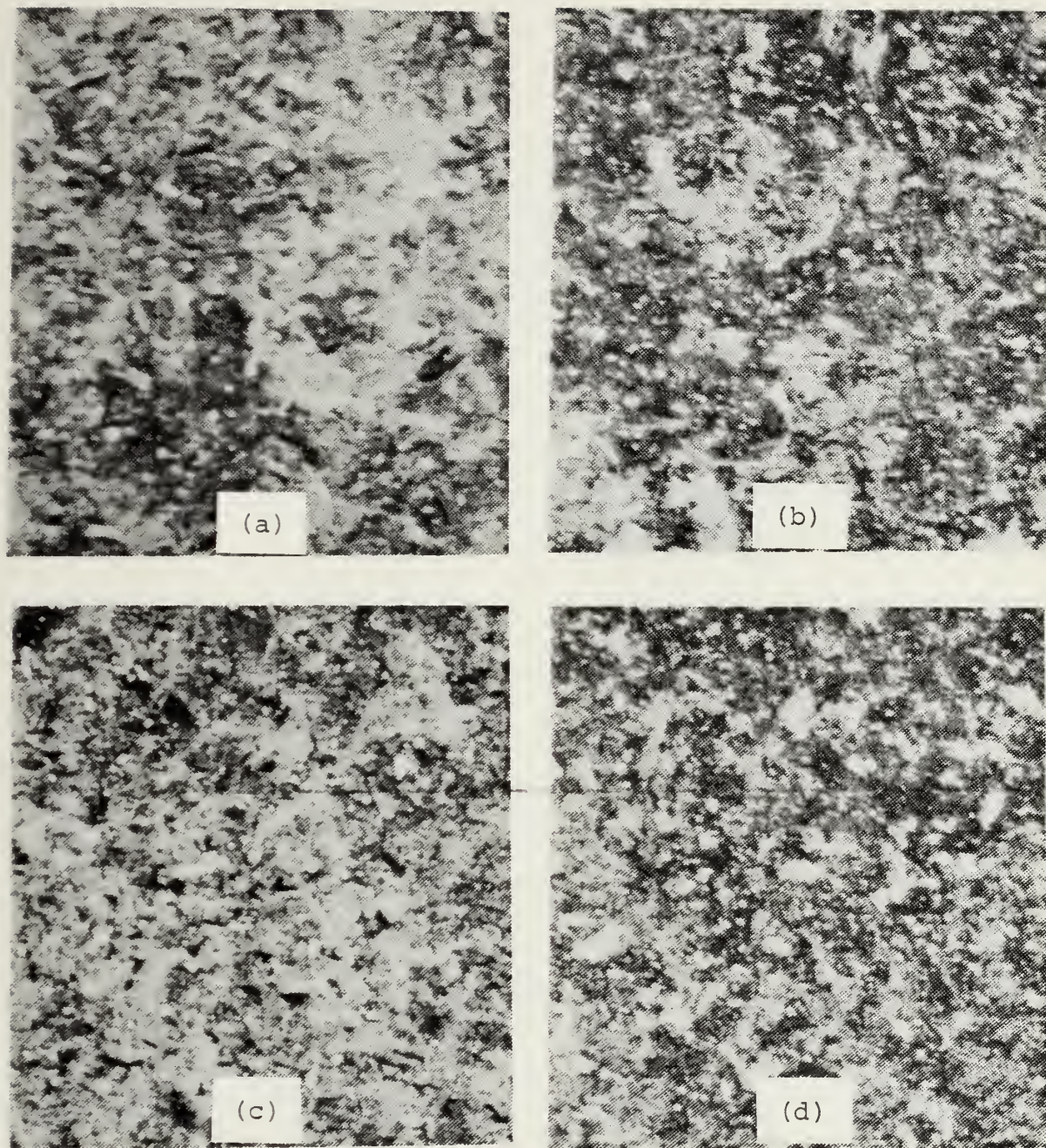


Figure 14. Optical micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 350°C. (a) AR after 20 seconds, (b) AR after 20 minutes, (c) HT-4 after 10 seconds, and (d) HT-4 after 20 minutes. Microstructures are (a & c) martensite and residual carbides and (b & d) bainite and residual carbides. Etched in 5% nital solution. 1600X.

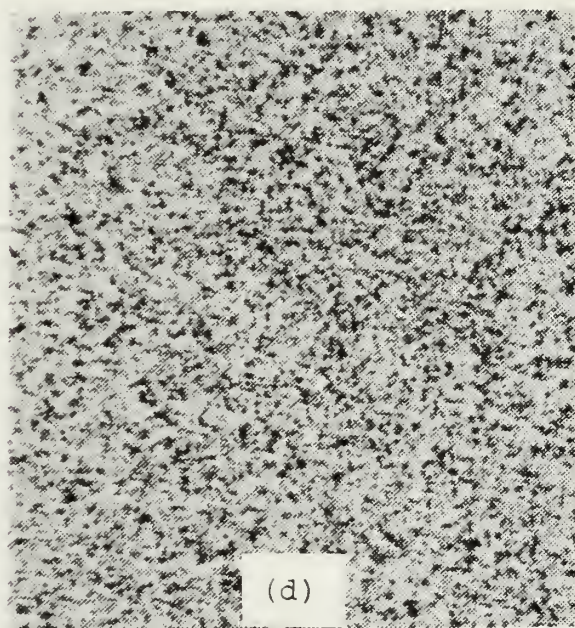
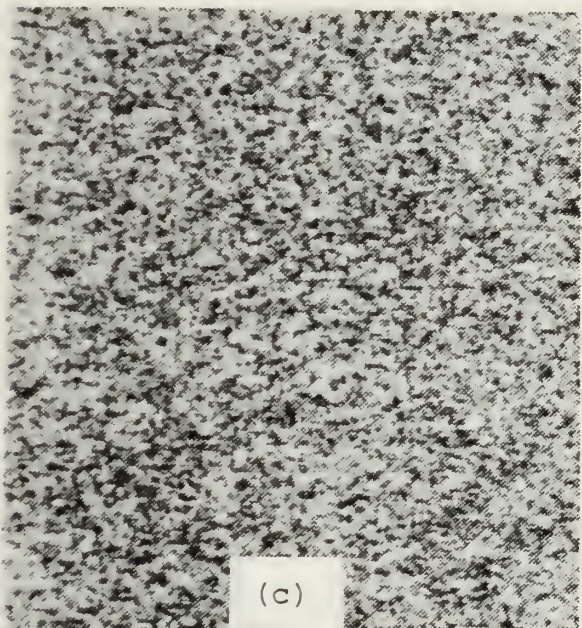
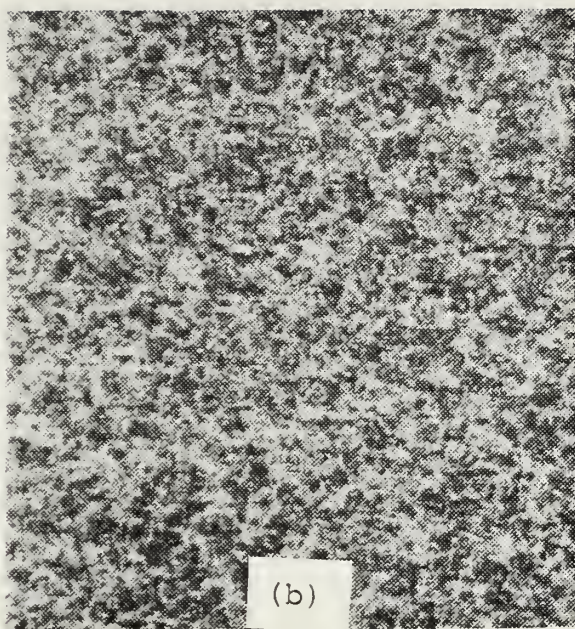
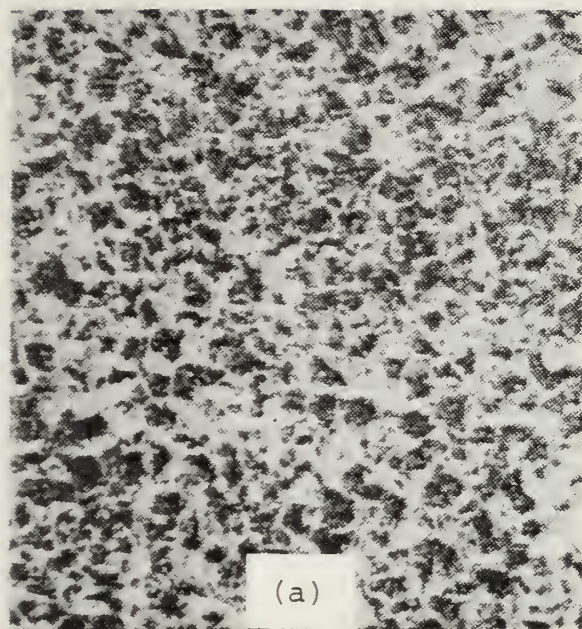


Figure 15. Optical micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 250°C. (a) AR after 20 seconds, (b) AR after 50 minutes, (c) HT-4 after 10 seconds, and (d) HT-4 after 50 minutes. The microstructure in (a & c) is martensite, in (b) is bainite and in (d) is a duplex martensite and bainite. Etched in 5% nital solution. 250X.

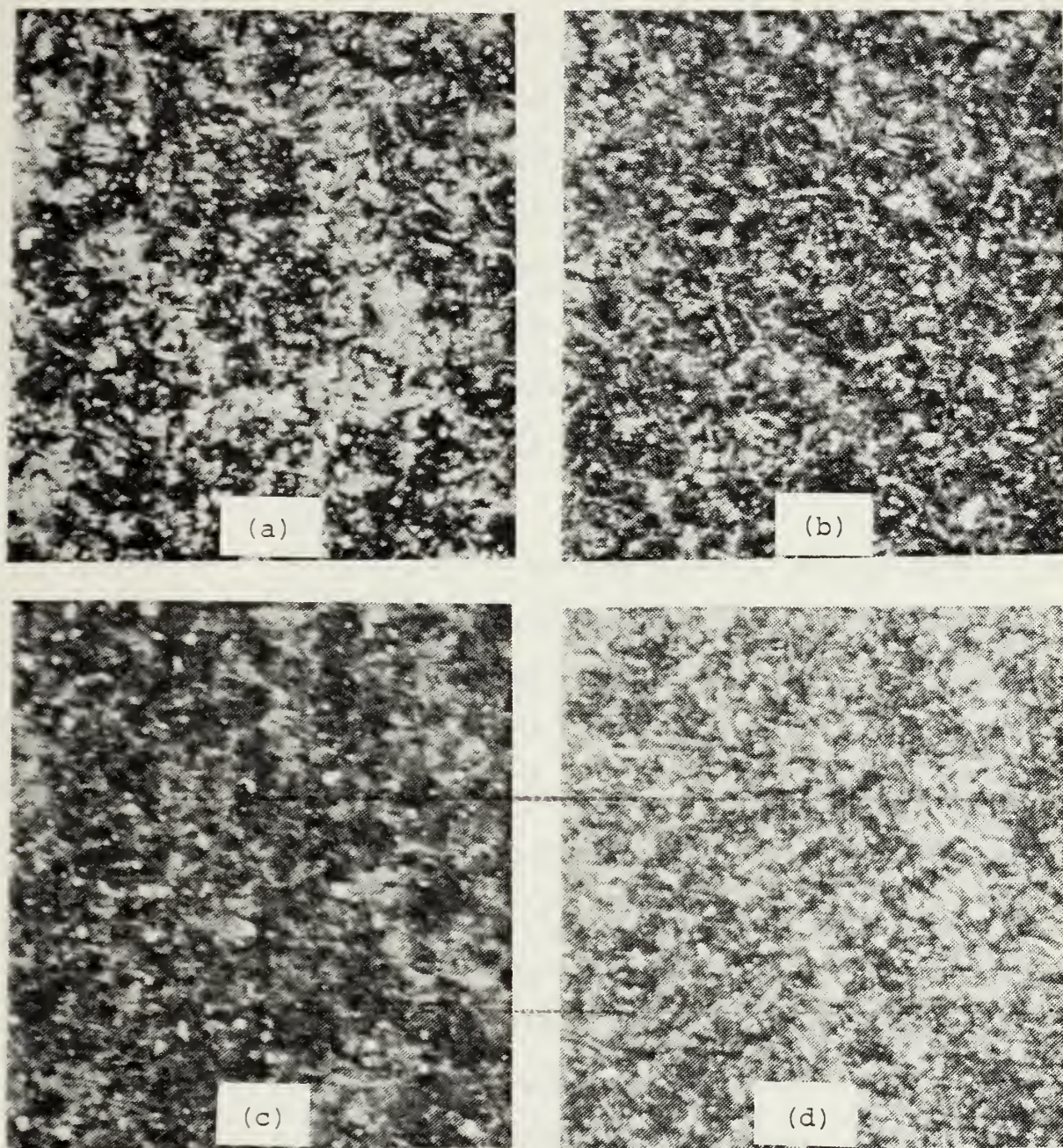


Figure 16. Optical micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 250°C. (a) AR after 20 seconds, (b) AR after 50 minutes, (c) HT-4 after 10 seconds, and (d) HT-4 after 50 minutes. Microstructures are residual carbides and martensite in (a & c), bainite in (b), and (d) is a duplex martensite and bainite. Etched in 5% nital solution. 1600X.

that the fine grain size of the thermomechanically refined material is retained throughout transformation and that, again, a coarsening of the refined residual carbides has taken place during austenitization. The microstructure in Figures 15d and 16d does appear to be a duplex martensite and bainite structure even for long transformation times. This would indicate that the martensite start temperature of the thermomechanically refined material is in the neighborhood of 250°C .

Carbide extraction transmission electron micrographs for the 350°C isothermal transformation heat treatment are shown in Figures 17a-c. Figures 17a and b are of the thermomechanically refined material and represent mid and completed points in the isothermal transformation reaction. These figures again clearly show that coarsening of the residual carbides has taken place and also show the progress of the bainite transformation by the increase in the dark feathery areas of the micrograph with increasing time of transformation. Figure 17c is the carbon extraction micrograph of the AR material after 20 seconds of isothermal transformation. The coarse residual carbides are similar in size to those present in the HT-4 material but the presence of bainite is not seen.

Carbide extraction transmission electron micrographs for the 250°C isothermal transformation heat treatment are depicted in Figures 18a-d. The carbide size and distribution

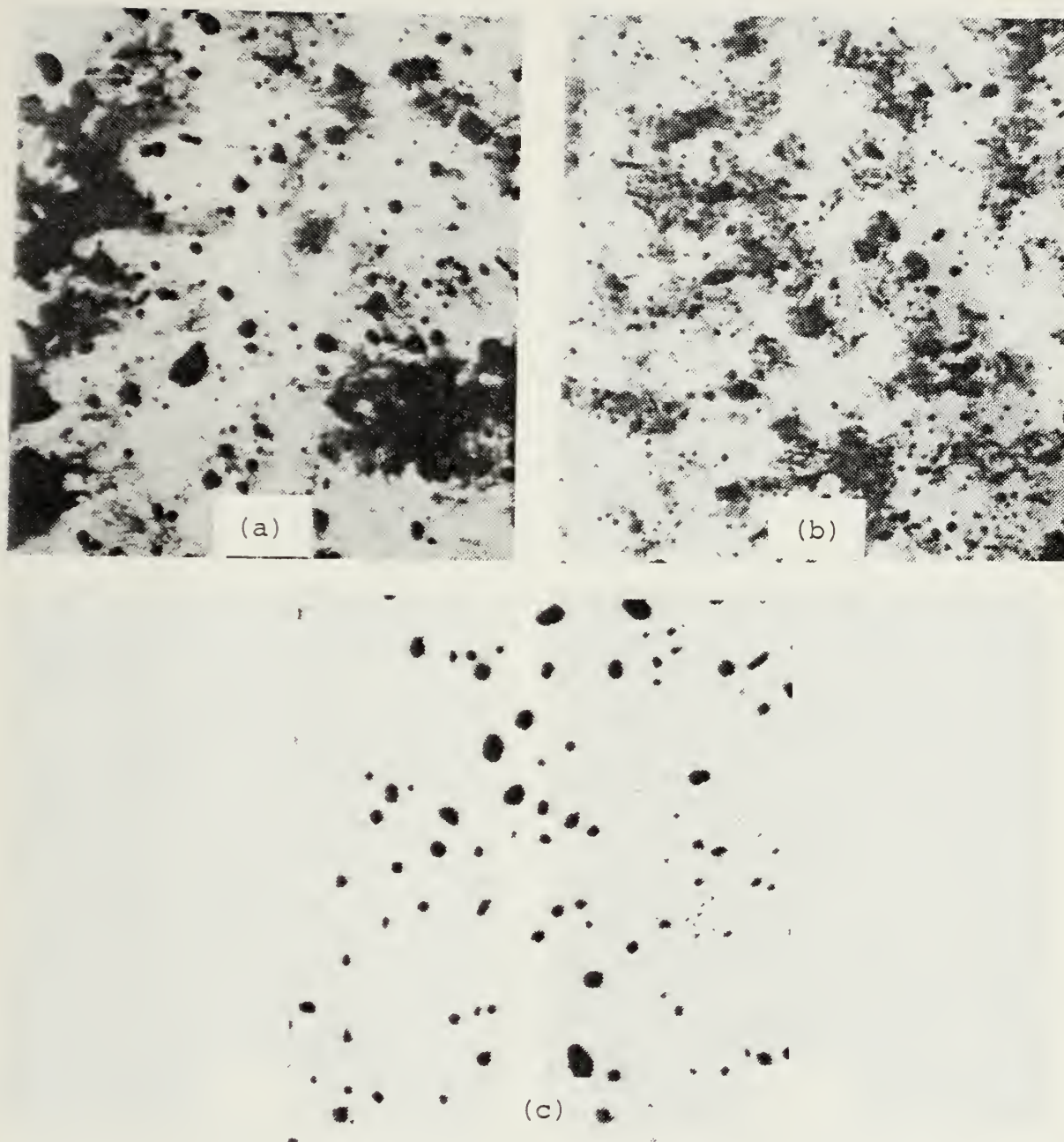


Figure 17. Carbide extraction replica micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 350°C. (a) HT-4 after 90 seconds, (b) HT-4 after 50 minutes, and (c) AR after 20 seconds. Microstructures show the coarsening of HT-4 carbides after austenitization and the dark feathery bainite. The AR microstructure is martensite with residual carbides. 5000X.

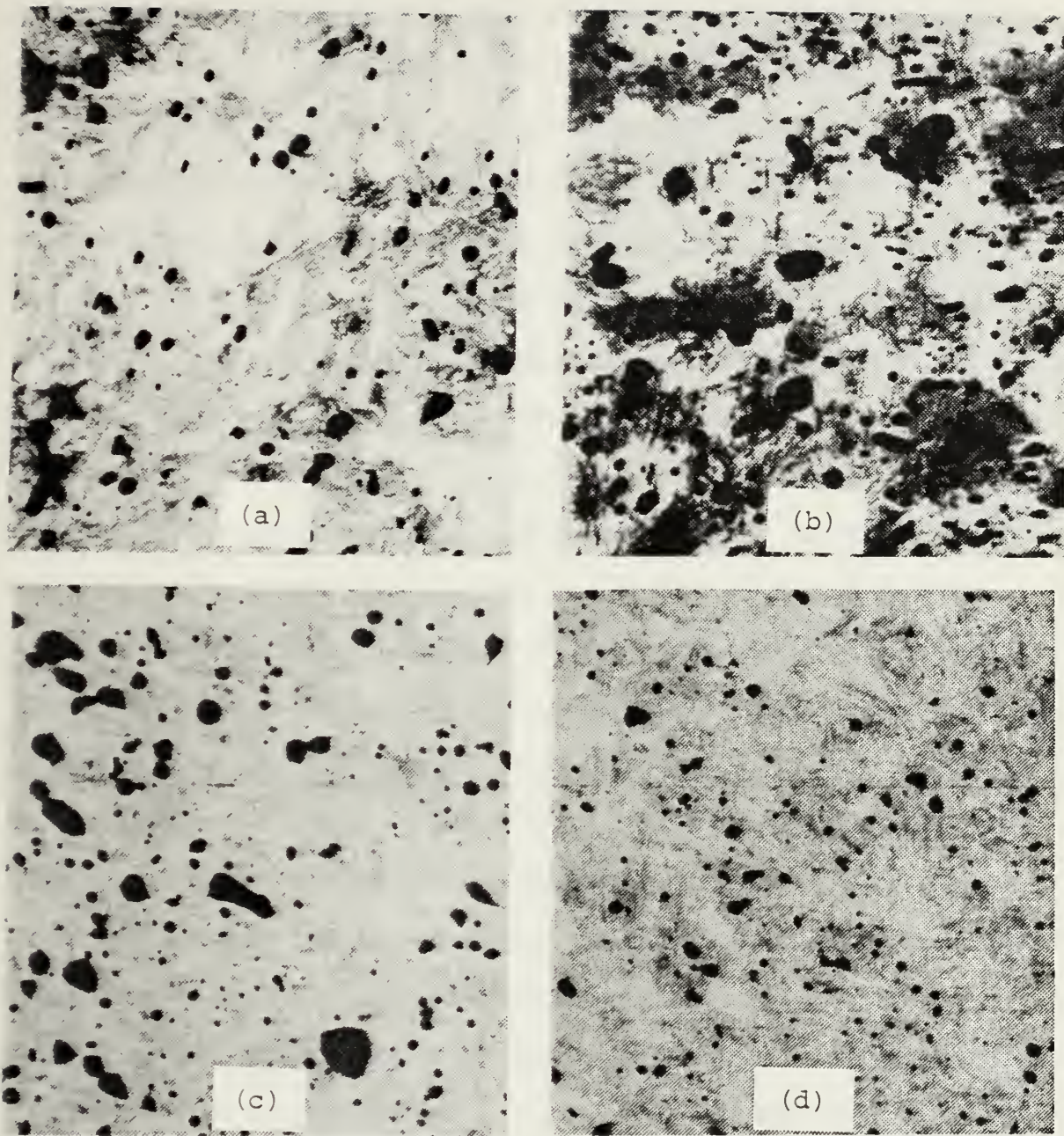


Figure 18. Carbide extraction replica micrographs of both the AR and HT-4 materials after 850°C austenitization and subsequent isothermal transformation heat treatment at 250°C. (a) AR after 10 minutes is duplex martensite and bainite, (b) AR after 50 minutes is bainite, (c) HT-4 after 10 seconds is martensite, and (d) HT-4 after 50 minutes is martensite. Residual carbides are evident throughout. 5000X.

for both the thermomechanically refined HT-4 material and the AR material are again seen to be very similar. A significant difference, however, is that the HT-4 material has no dark feathery areas indicative of bainite even for long transformation times. Instead, the microstructure appears to remain martensitic throughout transformation and this suggests again that the martensite start temperature, M_s , for the HT-4 material has been raised relative to that of the unprocessed AR material. Based on the I-T diagram of Figure 1 and these microstructural observations, M_s is near 250°C for the AR material, but above 250°C for HT-4. The next highest temperature examined, 275°C , indicated bainite in HT-4. At this point, then, the M_s temperature has been raised above 250°C for the thermomechanically processed material but is still below 275°C . This observation, that the M_s is raised by grain refinement of high carbon steel does not agree with the results of grain refinement in lower carbon steels [Ref. 13] and this factor would certainly apply to this higher carbon steel. However, the residual carbides may, as suggested earlier, be important in austenite decomposition as well as the austenite grain size. It is suggested that this factor, important in high-carbon steels, be given further evaluation.

D. RETAINED AUSTENITE

X-ray techniques previously described were employed to determine retained austenite content through the various

stages of isothermal transformation. Additionally, heat treated specimens were refrigerated to detect any retained austenite by change in hardness. The difference between the as-quenched, room temperature hardness and the refrigerated hardness is indicative of the presence of retained austenite. In all cases, this hardness change was insignificant and indicated that little or no austenite was retained during the isothermal transformation heat treatment.

The retained austenite content of both materials by x-ray diffraction methods was found to support the hardness test results of little or no retained austenite and the results of the X-ray analysis are presented in Table III. Figure 19 is the autographic record of the diffraction peaks obtained for the AR and HT-4 materials heat treated at 275°C for five and ten seconds, respectively. It can be seen that the (220) γ and (311) γ peaks expected at approximately 75° and 91° in 2 θ are not in evidence. Figure 20 presents the diffraction patterns obtained for the AR material heat treated at 250°C. In Figure 20a the transformation reaction period is 50 minutes and no retained austenite is seen. However, in Figure 20b the transformation period is only 20 seconds and here the (220) γ and (311) γ peaks are observed. From these peaks a 6% retained austenite content was calculated.

From Table III it is seen that at most only trace amounts (less than 2%) of austenite are retained in any of the 350°C and 275°C heat treatments. However, measurable amounts of

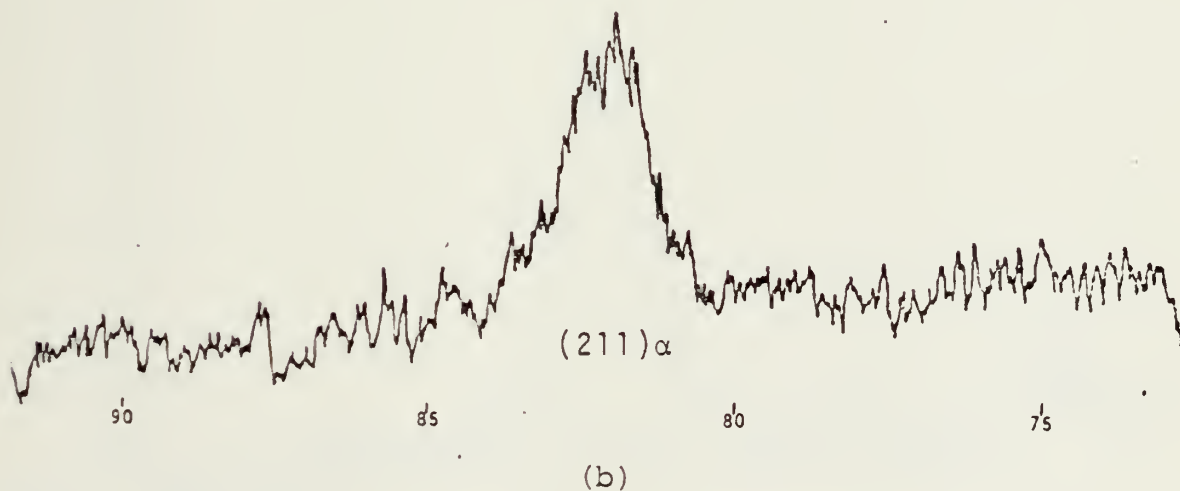
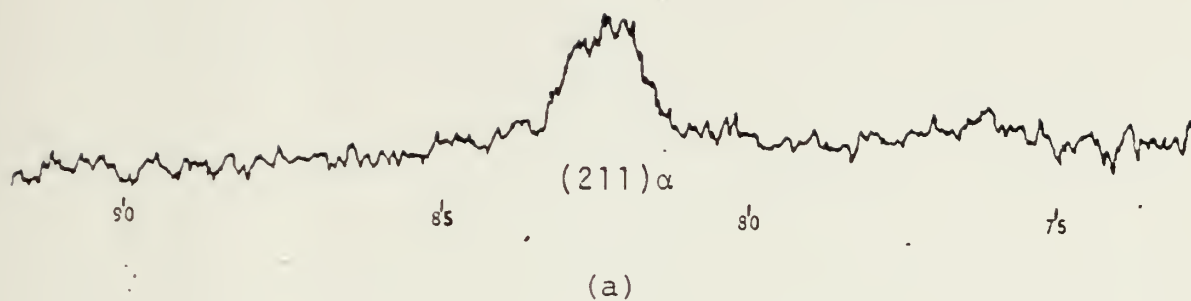


Figure 19. Autographic record of x-ray diffraction peaks for AR and HT-4 materials heat treated at 275°C. (a) Material AR after 5 seconds isothermal transformation. (b) Material HT-4 after 10 seconds of isothermal transformation. No retained austenite is evident.

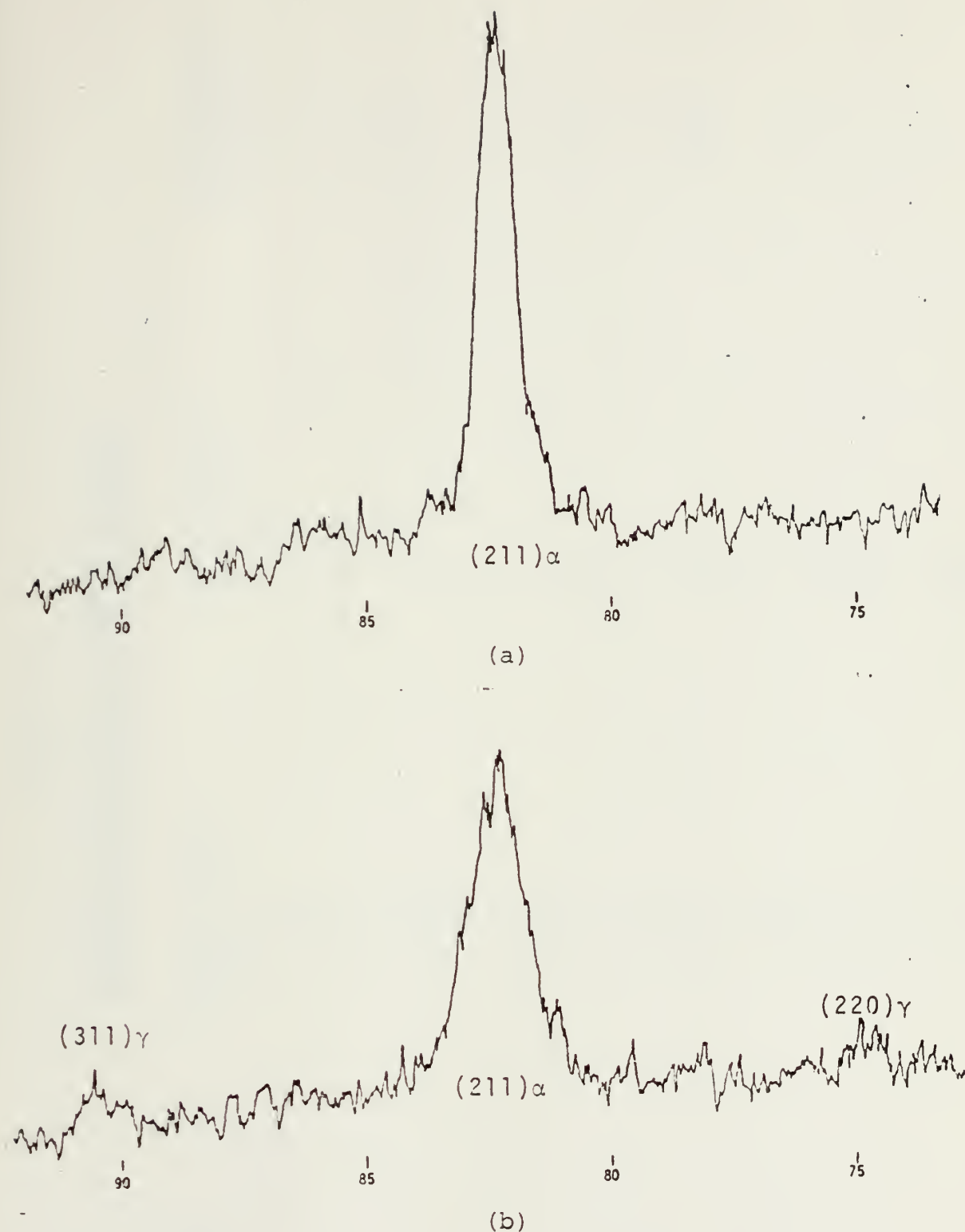


Figure 20. Autographic record of x-ray diffraction peaks for AR material heat treated at 250°C . (a) After 50 minutes of isothermal transformation, no retained austenite evident. (b) After 20 seconds of isothermal transformation, 6% retained austenite.

TABLE III

RETAINED AUSTENITE, VOLUME PERCENTAGE
AUSTENITIZED AT 850°C FOR 15 MINUTES

Specimen	Transformation Temp. (°C)	Transformation Time (sec)	Pct Retained Austenite
AR	350	20	0
AR	350	120	0
AR	350	1200	0
HT4	350	10	Trace
HT4	350	90	0
HT4	350	3000	0
AR	275	5	0
AR	275	600	Trace
AR	275	3000	0
HT4	275	10	0
HT4	275	300	Trace
HT4	275	3000	0
AR	250	20	6
AR	250	600	3
AR	250	3000	0
HT4	250	10	6
HT4	250	1200	7
HT4	250	3000	7

retained austenite are present within the 250°C heat treatment and retained austenite is seen to persist throughout the transformation of the HT-4 material at this temperature.

These results are surprising when compared to those of McCauley [Ref. 2] and Schultz [Ref. 3] in that they reported significant amounts of retained austenite in all heat treatments involving austenitization at similar temperatures. However, those previous studies generally involved direct quenching to ambient temperatures. The low amounts of retained austenite in the 350°C and 275°C heat treatments is thought to be a result of the interrupted quench procedure utilized and that even very short interrupted quench duration is sufficient to allow the unstable austenite to reduce its carbon content to a point where the martensite start and finish temperatures are raised. Thus, the unstable austenite may reach the martensite finish temperature at a point well above room temperature and the transformation will thus be complete at room temperature. At these low temperatures, nucleation is favored and fine carbide precipitates could be formed. Also, the mobility of the carbon atoms at these temperatures remains sufficient to allow the atoms to reach the residual carbides present and could result in some coarsening. In either case, the effect is to lower the matrix carbon content and raise the M_s and M_f temperatures, which may allow the austenite fully to transform to martensite at or above room temperature.

The HT-4 material heat treated at 250°C was found to have measurable amounts of retained austenite at all transformation times investigated. This is believed to be a direct result of the raised M_s temperature for the refined material. It is likely that grain refinement results in an increased number of nucleation sites for carbide precipitation and for this reason the HT-4 material will experience a larger increase in M_s temperature than the conventional AR material. In fact, such an effect would have to be sufficiently large to overcome the expected trend of suppression in M_s with grain size refinement [Ref. 13], although the role of residual carbides, in any event, is not clear.

The significance of this reduction in retained austenite by utilization of a short-duration interrupted quench above the M_s temperature is great. Kar, Horn and Zackay [Ref. 8] reported that in as-quenched and tempered 52100 steel the retained austenite underwent stress-induced transformation to untempered martensite. Such uncontrolled transformation of retained austenite is of serious concern as it leads to dimensional instability and is likely a factor in reduced toughness when it occurs. It is particularly undesirable in precision bearing applications. Thus, the reduction or elimination of retained austenite by such interrupted quench methods may allow for the manufacture of rolling element bearings with fully hardened, martensitic structures, yet without the presence of retained austenite. Note that,

in the original hardness data obtained in this research the hardness obtained following brief (10-20 seconds) quench interruptions was still Rc 65; these later results imply the absence of retained austenite in these materials. Finally, this result is independent of grain refinement and applies to the AR as well as HT-4 conditions of the material.

E. DIFFERENTIAL THERMAL ANALYSIS (DTA)

The results of the DTA performed are shown in Figure 21. These are tracings of autographic plots for the AR and HT-4 materials obtained with identical heating rates of 10 degrees Celsius per minute and show both the endothermic and exothermic peaks obtained by recording the power, required to maintain the heating rate, versus temperature. The plot for the AR material shows an endothermic peak beginning at approximately 770°C. This peak is associated with the spheroidal carbides of this material going into solution and the transformation of ferrite to austenite. It is seen that the endothermic reaction for the AR material is not complete until a temperature of 820°C is attained.

The plot for the thermomechanically refined HT-4 material also shows the endothermic reaction associated with the carbides dissolving and ferrite-to-austenite transformation. Here it is seen that the reaction begins at a lower temperature, 760°C, and that the peak is sharper and more narrow than for the AR material, indicating that the refined carbides

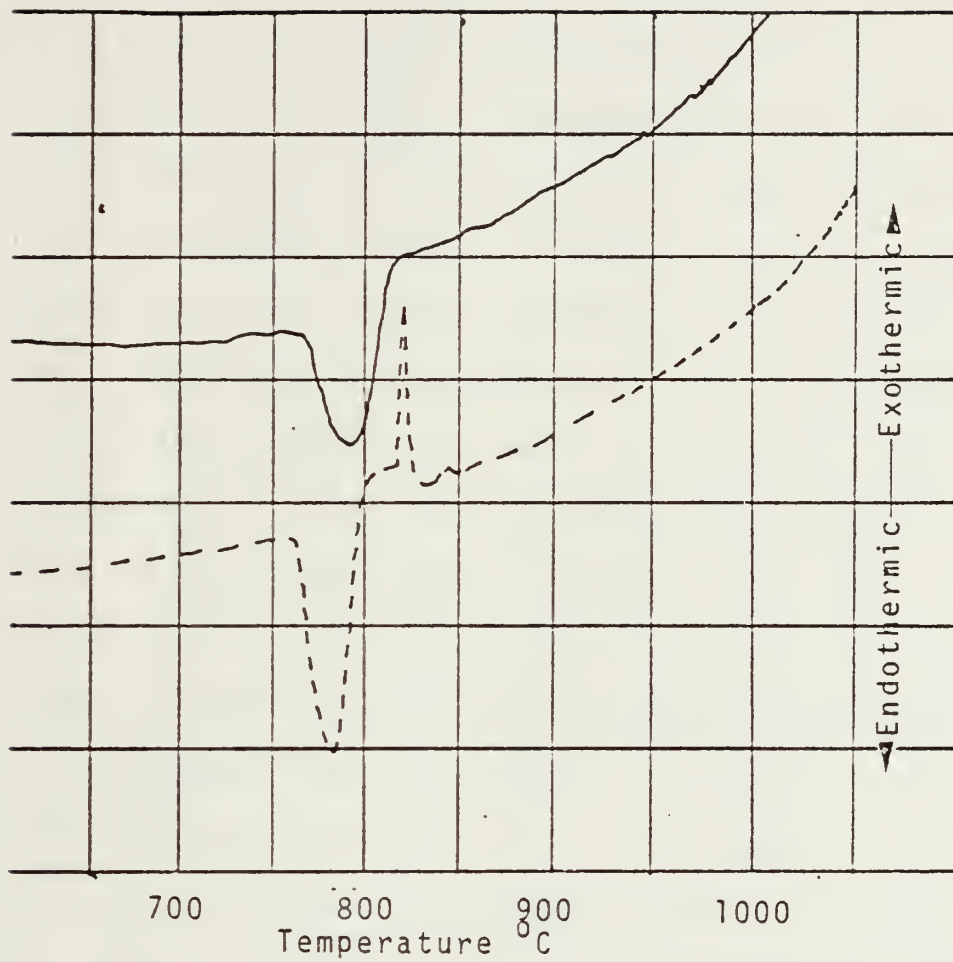


Figure 21. Autographic record of differential thermal analysis of AR and HT-4 materials.

are going into solution more rapidly and the ferrite-to-austenite transformation is accelerated. The endothermic reaction is complete by the time a temperature of 800°C is reached as compared to 820°C for the AR material. Additionally, two exothermic peaks not present in the AR plot are seen in the data for the HT-4 material. These exothermic peaks are of uncertain origin but may be associated with the coarsening of the refined carbides and the grain growth previously noted in the HT-4 material. It is believed that the first of these exothermic peaks is associated with the carbide coarsening. This peak is very sharp indicating the coarsening to take place very rapidly at about 820°C . The second, smaller peak is thought to be the result of grain growth and is seen to take place at approximately 840°C . It is also possible that the exothermic reactions are associated with the presence of W and the possible reaction to form WC or a W-containing carbide in these materials. This could result since the HT-4 thermomechanical process utilizes an austenitizing temperature of 1000°C , possibly sufficient to dissolve any such carbides originally present in the steel.

Analysis of this data, then, suggests that the HT-4 material should be austenitized at $800\text{--}810^{\circ}\text{C}$ to retain the carbide and grain refinement obtained during thermomechanical processing. It also suggests that a hardening treatment at this temperature does result in comparable matrix carbon

contents to that obtained in the AR material austenitized at much higher temperatures, due to the ease with which the refined carbides are dissolved into solution. Finally, this data supports the results of the microscopy examination in that both carbide coarsening and grain growth are likely to be seen in the HT-4 at temperatures below the 850°C austenitizing temperature used in this research.

F. TENSILE TEST RESULTS

Tensile tests were conducted in order better to understand the mechanical property differences between the standard, spheroidize annealed and thermomechanically processed HT-4 materials after isothermal transformation. The 350°C and 275°C isothermal transformation heat treatments were selected for comparison of the materials. Table IV contains the results of these tests.

After the 350°C isothermal transformation heat treatment, both materials are seen to be very similar in ultimate tensile strength, percent elongation and percent reduction in area to failure. In contrast, the results after the 275°C heat treatment vary greatly except for hardness. It is seen that the thermomechanically processed HT-4 material, while slightly lower in ultimate tensile strength, does exhibit significantly greater elongation and reduction in area to failure. This indicates that the 275°C isothermal transformation heat treatment results in a

TABLE IV

MECHANICAL PROPERTIES OF ISOTHERMALLY TRANSFORMED MATERIAL AUSTENITIZED AT 850°C FOR 15 MINUTES

Specimen	Transformation		Ultimate Tensile Strength MPa (Ksi)	% Elongation	% Reduction in Area	Hardness Rc
	Temp. (°)	Time, Min.				
AR	350	20	1613.4 (234)	10	30	47
HT4	350	20	1613.4 (234)	12	32	47
AR	275	50	2247.8 (326)	4	3	58
HT4	275	50	2096.1 (304)	10	24	58

material of suitable hardness for bearing applications [Ref. 9], and that this material possesses the desirable characteristic of greater ductility indicative of a higher toughness at this hardness and strength level. The trend noted is consistent with that reported by Chung [Ref. 1] and McCauley [Ref. 2] and confirms their results. The enhanced ductility must be a result of the grain size refinement demonstrated for this material.

IV. CONCLUSIONS AND RECOMMENDATIONS

Based on the experimental observations and results the following conclusions are drawn:

1. The isothermal transformation characteristics of the thermomechanically processed HT-4 material are essentially identical to those of the AR material.

2. The grain size of the thermomechanically refined HT-4 material is retained throughout austenitization at 850°C and throughout subsequent isothermal transformation heat treatment.

3. The carbide refinement obtained during thermomechanical processing was not retained in the material utilized during austenitization at 850°C and the residual carbides present after heat treatment are comparable in size to those of the heat treated AR material.

4. The martensite start temperature for the thermomechanically processed HT-4 material was found to be above 250°C but below 275°C .

5. Austenitization of AR and HT-4 materials at 850°C with interrupted quenching or isothermal transformation above the martensite start temperature results in little or no retained austenite in either material after oil quenching to room temperature.

6. The thermomechanically processed HT-4 material should be austenitized at 800-810°C to retain the carbide and grain refinement obtained during the thermomechanical processing.

7. The rate of carbide dissolution during austenitizing of the HT-4 material is greater than that for the AR material and the carbides are dissolved at lower temperatures.

8. The 850°C austenitization of the HT-4 material and subsequent isothermal transformation heat treatment at 275°C results in a material possessing hardness, strength and ductility suitable for use as a high strength and toughness rolling element bearing material.

Recommendations for further research are:

1. Study of the effect of austenitizing temperature upon the refined carbides in thermomechanically processed materials.

2. Study of the fracture toughness characteristics of thermomechanically processed materials after isothermal transformation heat treatment above the martensite start temperature.

3. Research on the interrupted quench above the martensite start temperature and its effect on retained austenite and the effect of austenitizing temperature on these observations.

4. Differential thermal analysis of 52100 steel with various Tungsten contents to assess its effect on carbide coarsening.

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